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ANALYTICAL ABSTRACTS

JOURNALS

The following journals and publications were abstracted regularly during 1954, but abstracts from journals other than these have also been published from time to time.

Acta Medica Scandinavica.
Acta Pharmacologica et Toxicologica. København.
Acta Physiologica Scandinavica.
Ätherische Öle, Riechstoffe, Parfümerien, Essenzen und Aromen.
Agra University Journal of Research.
American Dyestuff Reporter.
American Ink Maker.
American Journal of Clinical Pathology.
American Journal of the Medical Sciences.
American Paint Journal.
Anales de Bromatologia.
Anales de la Real Sociedad Española de Física y Química.
Analytica Chimica Acta.
Analytical Chemistry.
Analyst.
Angewandte Chemie.
Annales de Chimie, Paris.
Annales de Falsifications et des Fraudes, Paris.
Annales de la Nutrition et de l'Alimentation.
Annales Pharmaceutiques Françaises.
Annali di Chimica Applica, Roma.
Antibiotics and Chemotherapy.
Applied Spectroscopy.
Aptechnoe Delo.
Archiv für Mikrobiologie, Berlin.
Archives of Biochemistry and Biophysics.
Archives of Industrial Hygiene and Occupational Medicine, Baltimore.
Archiv för Kemi, Stockholm.
Australian Journal of Agricultural Research.
Australian Journal of Applied Science.
Australian Journal of Chemistry.

Bericht der deutschen keramischen Gesellschaft. Berlin.
Biochemical Journal.
Biochemische Zeitschrift.
Biochimica et Biophysica Acta.
Biometrics. Washington.
Bitumen, Teere, Asphalte, Peche.
Boletin de Radiactividad.
Bolletino Chemico-Farmaceutico.
Brauerei.
Brauwissenschaft.
Brennstoff-Chemie.
British Journal of Applied Physics.
British Journal of Pharmacology and Chemotherapy.
British Medical Bulletin.
British Medical Journal.
Bulletin de l'Académie Polonaise des Sciences.
Bulletin of the American Society for Testing Materials.
Bulletin de l'Association Française des Chimistes des Industries de Cuir et Documents Scientifiques et Techniques des Industries du Cuir.
Bulletin of the Chemical Society of Japan.
Bulletin de l'Institut Agronomique et des Stations de Gembloux.
Bulletin of the "Boris Kidrich" Institute of Nuclear Sciences. Belgrade.
Bulletin of the Institution of Mining and Metallurgy.
Bulletin of the Research Council of Israel.
Bulletin. Société Chimique de Belgique.
Bulletin de la Société de Chimie Biologique.
Bulletin. Société Chimique de France.

Canadian Chemistry and Process Industries.
Canadian Journal of Agricultural Sciences.
Canadian Journal of Biochemistry and Physiology.
Canadian Journal of Chemistry.
Canadian Journal of Technology.
Cereal Chemistry.
Chemical and Engineering News.

Chemical Products.
Chemické Listy pro Vědu a Průmysl. Praha.
Chemie - Ingenieur - Technik.
Chemikerzeitung.
Chemisch Weekblad.
Chemist-Analyst.
Chemist and Druggist.
Chemistry and Industry.
Chimia.
Chimica e l'Industria.
Chimie Analytique.
Chimie et Industrie.
Chimie des Peintures.
Collection of Czechoslovak Chemical Communications.
Colonial Plant and Animal Products.
Combustibles.
Comptes Rendus Hebdomadaires des Séances de l'Académie d'Agriculture de France.
Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Paris.
Contributions. Boyce Thompson Institute for Plant Research.
Current Science.

Dansk Tidsskrift for Farmaci. Kjøbenhavn.
Deutsche Apothekerzeitung.
Deutsche medizinische Wochenschrift.
Dissertation Abstracts.
Doklady Akademii Nauk, SSSR. [Compt. Rend. Acad. Sci., U.S.S.R.]

Electroplating.
Endocrinology. Glendale. Calif.
Endokrinologie. Leipzig.
Erdöl und Kohle.
Experientia.

Farmaceutisk Revy. Stockholm.
Faserforschung und Textiltechnik.
Fibres.
Finnish Paper and Timber Journal.
Fuel, London.

Gazzetta Chimica Italiana.

Helvetica Chimica Acta.
Hoppe-Seyler's Zeitschrift für physiologische Chemie.
Hutnické Listy.

India Rubber World.
Indian Journal of Dairy Science.
Indian Journal of Medical Research.
Industria della Vernice.
Industrial Chemist and Chemical Manufacturer.
Industrial and Engineering Chemistry.
Industrie Chimique Belge.
Industries Agricoles et Alimentaires.
Información de Química Analitica.
Institute of Petroleum Review.
Instruments and Automation.
International Archives of Allergy and Applied Immunology.
Iowa State College Journal of Science.
Izvestiya Akademii Nauk SSSR, Otdelenie Khimicheskikh Nauk. Moscow. [Bull. Acad. Sci., U.S.S.R.]

Journal of Agricultural and Food Chemistry.
Journal of the American Chemical Society.
Journal of the American Leather Chemists' Association.
Journal of the American Oil Chemists' Society.
Journal of the American Pharmaceutical Association.
Journal of the American Water Works Association.
Journal of Applied Chemistry, London.
Journal of the Association of Official and Agricultural Chemists.
Journal of Biological Chemistry.
Journal of Chemical Education.
Journal of the Chemical, Metallurgical and Mining Society of South Africa.
Jounal of the Chemical Society, London.
Journal de Chimie Physique.
Journal of Clinical Investigation. Baltimore.

Journal of Clinical Pathology.
Journal of Dairy Science.
Journal of Economic Entomology.
Journal of the Electrochemical Society.
Journal of the Indian Chemical Society.
Journal of the Indian Institute of Science.
Journal of the Institute of Brewing.
Journal of the Institute of Fuel.
Journal of the Institute of Petroleum.
Journal of the Institution of Chemists, India.
Journal of the Iron and Steel Institute.
Journal of Laboratory and Clinical Medicine.
Journal of Medical Laboratory Technology.
Journal of the New England Water Works Association.
Journal of the Optical Society of America.
Journal de Pharmacie de Belgique.
Journal of Pharmacy and Pharmacology.
Journal of Physical Chemistry.
Journal of Research and Development.   British Cast Iron Research Association.
Journal of Research of the National Bureau of Standards.
Journal of the Rubber Research Institution of Malaya.
Journal of the South African Chemical Institute.
Journal of the Science of Food and Agriculture.
Journal of Scientific and Industrial Research, India.
Journal of Scientific Instruments.
Journal of the Society of Dyers and Colourists.
Journal of the Society of Glass Technology.
Journal of the Society of Leather Trades' Chemists.
Journal of the Textile Institute.   Manchester.

Klinische Wochenschrift.
Kolloidzeitschrift.
Kungliga Lantbruks-Högskolans Annaler.
Kunststoffe.   Zeitschrift für Erzeugung und Verwendung veredelter oder chemisch hergestellter stoffe.
    München.

Laboratory Practice.
Lancet.

Medical Journal of Australia.
Metal Finishing.
Metallurgia.
Microchimica Acta.
Ministry of Fuel and Power Safety in Mines Research Reports.
Mitteilungen des chemischen Forschungs-Instituts der Industrie.   Wien.
Mitteilungen aus dem Gebiete Lebensmitteluntersuchungen und Hygiene.
Mitteilungen der Höheren Bundeslehrund Versuchanstalt für Wein-und Obstbau.   Klosterneuberg.
Mitteilungen der Versuchsanstalt für Gärungsgewerbe und des Instituts für angewandte Mikrobiologie.
Monitore Farmaceutico dell'Agenzia Leoni e De Lorenzo.

Nature.
Naturwissenschaften.
New Zealand Journal of Science and Technology.

Official Digest of the Federation of Paint and Varnish Production Clubs.
Olii Minerali, Grassi e Saponi, Colori e Vernici.

Paint Industry Magazine.
Paintindia.
Paper Maker and British Trade Journal.
Peintures - Pigments - Vernis.
Perfumery and Essential Oil Record.
Pharmaceutical Journal and Pharmacist.
Pharmaceutisch Weekblad voor Nederland.
Prace Instijtito Ministerstwa Hutnictwa.
Praktische Chemie.
Proceedings of the American Society for Testing Materials.
Proceedings of the Indian Academy of Sciences.
Proceedings of the Mayo Clinic.
Proceedings of the Society for Experimental Biology and Medicine.

Receuil des Travaux Chimiques des Pays-Bas.
Reports of the Government Chemical Industrial Research Institute, Tokyo.
Research.

Review of Coal Tar Technology.
Revista de Ciencia Aplicada.
Revista de le Sociedad Venezolana de Química.
Review of Scientific Instruments.
Revue de l'Institut Français du Pétrole et Annales des Combustibles Liquides.
Ricerca Scientifica.
Rocznik Chemji.

Schweizerische medizinische Wochenschift.
Science.
Sewage and Industrial Wastes.
Shirley Institute Memoirs.
Soap, New York.
Soil Science.
South African Industrial Chemist.
Spectrochimica Acta.
Sugar. N.Y.
Svensk Papperstidning.

Technology Reports of the Tôhoku Imperial University.
Transactions of the American Association of Cereal Chemists.
Transactions of the British Ceramic Society.

Verfkroniek.

Water and Sewage Works.

Zeitschrift für analytische Chemie.
Zeitschrift für Pflanzenernährung und Düngung.
Zhurnal Analiticheskoy Khimii, Moscow. [J. Anal. Chem., U.S.S.R.]
Zhurnal Obshchey Khimii, Moscow. [J. Gen. Chem., U.S.S.R.]
Zhurnal Prikladnoy Khimii, Moscow. [J. Appl. Chem., U.S.S.R.]

ACKNOWLEDGEMENTS

ERRATA

Abstract No.	Line	
27	5	*for* Y. J. Webber *read* T. J. Webber.
89	2	*for* Lyndersen *read* Lydersen.
111	1	*for* Spectrophometric *read* Spectrophotometric.
573	7	*for* saponifiable *read* unsaponifiable.
628	3	*for* Waligóta *read* Waligóra.
729	8	*for* Dodecanal *read* Dodecanol.
749	14	*for* 732 *read* 731.
753	20	Delete the whole line from "lead" to "given."
758	24	The first word should be "involving."
788	3	*for* Frohmann *read* Frohman.
792	28	*for* chichonine *read* cinchonine.
1981	3	*for* Zochrow *read* Zodrow.
2190	6	*for* taurocyanine *read* taurocyamine.
2369	14	*for* Yt *read* Y.
2583	1	*for* fiame *read* flame.
2673	7	*for* 1 to 1 *read* 1 to 2.
2929	12	*for* 20 *read* 200.
2967	2	*for* zinc *read* tin.
	10	*for* acetic *read* tartaric.
3016	3	*for* I. Bertetti *read* J. Bertetti.

ANALYTICAL
ABSTRACTS

Dealing with all branches
of Analytical Chemistry:
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Chemistry

ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

1. **New method for the qualitative analysis of anions.** C. Malen and P. Bevillard (*Anal. Chim. Acta*, 1953, **8** [6], 493–509).—The proposed system is developed from that of Engelder ("Elementary Qualitative Analysis," Second Edition, New York, John Wiley & Sons, Inc., 1933). The anions are separated into 7 groups based on the insolubility of Li, Ca, Ba, Zn, and Pb salts. The first group contain CO_3'', NO_3', ClO', BrO', CH_3COO'. Distinguishing tests and confirmatory tests are given for each anion. The second group is pptd by $LiNO_3$, and consists of F', SiO_3'', PO_4''', AsO_4''' and CO_3''. The decoloration of alizarin complexes with Zr is used for F'. Group III contains C_2O_4'', IO_3', MoO_4'', WO_4'', AsO_3''', SO_3'' and BO_2', all of which are pptd. by Ca from boiling solution. These are divided into two sub-groups by 20 per cent. CH_3COOH. Mo is detected by forming a molybdothiocyanate with a violet-red colour and confirmed by phosphomolybdate. Group IV is pptd. by boiling with $Ba(NO_3)_2$ solution and contains CrO_4'', SiF_6'', SO_4'', S_2O_3'', BrO_3', and $Fe(CN)_6'''$. Group V is CN', $Fe(CN)_6''''$, $Fe(CN)_6'''$ and S'', all of which are pptd. by $Zn(NO_3)_2$ in the cold. Group VI contains halides, SCN', and formates which are pptd. by $Pb(NO_3)_2$. Group VII containing anions whose lead salts are soluble, MnO_4', H_2PO_2', NO_2', ClO_3', CH_3COO', NO_3'. Some duplication is admitted in that anions which would be eliminated in earlier groups are also placed in later groups by their chemical properties. In using the complete scheme these are dealt with in the first group in which they occur. Incompatibility of certain anions (*e.g.*, $Fe(CN)_6''''$ and S'') simplifies use in some instances but special action is needed for others (*e.g.*, SiO_3'' and F'). W. C. WAKE

2. **Systematic approach to the choice of organic reagents for metal ions.** R. J. P. Williams (*Analyst*, 1953, **78**, 586–594).—Choice of an organic reagent for a particular metal ion is limited by the nature of the free energy change occurring on formation of the complex in aq. solution. A broad division of such reactions is made, based upon whether the entropy change or the heat change is the more important in the free energy change. Examples of the successful use of organic reagents are examined and suggestions are made for design of new reagents. A. O. JONES

3. **Use of dithizone as extraction indicator.** A. G. Karabash (*J. Anal. Chem., U.S.S.R.*, 1953, **8**, 140–151).—Titrations are based on reactions involving metal dithizone complexes. The aq. solution together with 1/20th or 1/10th its vol. of $\simeq 0.0001$ or 0.00005 M dithizone in carbon tetrachloride, chloroform, or benzene is titrated with a suitable solution until a definite colour change appears in the organic solvent layer. Towards the end of the titration each addition is followed by shaking for 5 to 10 sec. The end-point is indicated when a colour change produced does not disappear in 30 sec. shaking and the colour remains unchanged on addition of 1 to 2 drops of the indicator solution. Examples are—titration of $Ag^{\cdot}$ in 1.0 to 9.0 N H_2SO_4 with KI giving an end-point accurate to 0.01 ml of 0.002 M KI; titration of $Ag^{\cdot}$ with 0.01 N KBr in 4 N H_2SO_4, and similarly with 0.01 N KCNS at a H_2SO_4 concn. of 3.5 N; titration of I', Br', and CNS' with $Ag^{\cdot}$ under the above-mentioned conditions; determination of Cl' by addition of $AgNO_3$, filtration, and titration of excess $Ag^{\cdot}$ by means of KI; determination of CN' (*a*) by titration in weakly alkaline solution, 0.3 to 0.6 N aq. NH_3 or 0.1 to 0.2 N NaOH, with $AgNO_3$ in presence of 0.01 per cent. dithizone solution in carbon tetrachloride, or (*b*) use of Pb dithizonate, the solution in 0.3 to 0.6 N aq. NH_3 being treated with a drop of 0.025 M $Pb(NO_3)_2$ and 1 to 2 ml of 0.01 per cent. dithizone and titrated with $AgNO_3$, the end-point being sensitive to 0.01 to 0.02 ml of 0.025 N $AgNO_3$; determination of $Hg^{\cdot\cdot}$ in $(1 + 3)$ H_2SO_4 by titration with 0.02 to 0.002 M KI; determination of $Au^{\cdot\cdot\cdot}$ in 0.5 to 1.0 N HCl saturated with NaCl or KCl with 0.01 to 0.002 N Na thiosulphate; determination of heavy metals by titration with Na_2S (*a*) titration of $Pb^{\cdot\cdot}$ and $Cd^{\cdot\cdot}$ in H_3BO_3 solution by means of 0.01–0.005 M Na_2S in presence of dithizone in benzene (*b*) titration of $Bi^{\cdot\cdot\cdot}$ in $NaHCO_3$ solution with 0.005 M Na_2S in presence of dithizone in benzene or carbon tetrachloride, (*c*) titration of $Hg^{\cdot\cdot}$ and $Ag^{\cdot}$ in dil. H_2SO_4 solution; determination of S'' by titration with a heavy metal salt under similar conditions. G. S. SMITH

4. **Paper chromatography of inorganic cations with dithizone.** G. Venturello and A. M. Ghe (*Ann. Chim., Roma*, 1953, **43**, 267–272).—Inorganic cations ($Ag^{\cdot}$, $Pb^{\cdot\cdot}$, $Cu^{\cdot\cdot}$, $Zn^{\cdot\cdot}$, $Cd^{\cdot\cdot}$, $Hg^{\cdot\cdot}$, $Mn^{\cdot\cdot}$, $Ni^{\cdot\cdot}$ $Co^{\cdot\cdot}$ and $Bi^{\cdot\cdot\cdot}$) are separated by chromatography in aq. phenol on paper impregnated with dithizone (diphenylthiocarbazone). The mixtures $Co^{\cdot\cdot}$ - $Ni^{\cdot\cdot}$ and $Ag^{\cdot}$ - $Hg^{\cdot\cdot}$ - $Bi^{\cdot\cdot\cdot}$ are not separated. The method is sensitive to 0.05 μg or less, and will detect metals in the presence of excess of other cations. J. K. LANDQUIST

5. **Precipitation phenomena in gravimetric determinations with cupferron.** E. Gastinger (*Z. anal. Chem.*, 1953, **139** [1], 1–14).—A detailed account of the pptn. phenomena that occur when metals are pptd. with cupferron is given. Special reference is made to Al and Zn and to the best conditions for pptn. The effect of free nitrosophenylhydroxylamine on the pptn. is studied, and it is due to the formation of this compound that sharp separation of metals by cupferron is not possible. The practical conclusions reached are that good separations are possible at low acidities by keeping the vol. low and by using small quantities of metal. H_2SO_4 should always be used and a saturated solution of cupferron used for washing. The temp. should not exceed $10°$ C. G. P. COOK

6. 2 : 4 : 7-Trinitrofluorenone as reagent for microscopic fusion analysis. Application to polynuclear aromatics and some substituted benzenes. D. E. Laskowski, D. G. Grabar and W. C. McCrone (*Anal. Chem.*, 1953, **25** [9], 1400–1404).—An addition compound is formed with 2:4:7-trinitrofluorenone by the Kofler microscopic mixed fusion technique. By means of a simple microscope with a Kofler hot stage, the m.p. of the unknown, the addition compound, and the two eutectics formed are determined and the data obtained provide the means of positive identification of polynuclear compounds. The melting point and fusion data for 30 polynuclear aromatics and photomicrographs of the mixed fusion preparations for each compound are given.   G. P. COOK

7. Instability of diethyldithiocarbamic acid at low pH. A. E. Martin (*Anal. Chem.*, 1953, **25** [8], 1260–1261).—In the investigation of the irregular and low recoveries of copper as a complex of diethyldithiocarbamate with chloroform from citrate buffers at pH 1 to 6, the influence of pH on the extraction, extraction of reagent into solvent, and stability of reagent with time were determined. Results are reported and discussed.   O. M. JONES

8. Differential mano- and volumetric methods. B. Kok, G. W. Veltkamp and W. P. Gelderman (*Biochim. Biophys. Acta*, 1953, **11**, 7–16).—The movement of fluid columns in glass capillaries was studied in order to obtain more insight into the restrictions introduced by their use as indicators in manometric and volumetric apparatus. It appears that these restrictions mainly concern the limitation of the speed of response, but the influence of other factors is discussed. Simple but accurate apparatus is needed for kinetic experiments in photosynthesis.   V. M. WOOTTON

9. Gradient elution analysis. I. General treatment. R. S. Alm, R. J. P. Williams and A. Tiselius (*Acta Chem. Scand.*, 1952, **6** [6], 826–836).—A new general procedure for the chromatographic analysis of mixtures of compounds is outlined. A continuously changing eluting medium is produced (external to the column) in a mixing chamber. In operating this "gradient elution method," factors such as the spreading of zones, the separation of components, and the pretreatment of the adsorbent are discussed. Spreading of zones normally occurs with substances giving curved adsorption isotherms, but if the concn. of eluent is linearly increased the initial spreading is followed by a contraction of the zone; finally the zone travels as an almost symmetrical elution peak as the isotherm becomes linear. The zone-spreading is still controlled by the gradient in which the peak travels; with steep gradients, zone development occurs rapidly and the use of excessively long columns is avoided. Good separations of oligosaccharides, fatty acids, sterols, peptides, and aminoacids are already achieved and subsequent experiments show further widespread applications of the method.   D. R. GLASSON

10. Continuous extraction of composite substances according to the volume-replacement principle with change of one parameter. R. K. Zahn and Irmgard Stahl (*Hoppe-Seyl. Z.*, 1953, **293** [1], 1–10).—A procedure for the continuous extraction of composite substances of general application, but particularly suitable for proteins, requiring very simple apparatus is described; an example is given of its application to the ammonium sulphate fractionation of a yeast autolysate. Mathematical treatment of

parameter change on the basis of the volume-replacement principle is given. A ppt. of the substance is formed on a suitable carrier substance (kieselguhr, SiO₂-gel) and eluted with a solution of continuously changing concentration, whereby components are brought into solution one after another and collected as separate fractions.
        W. MANZ

11. A general review of some recent developments concerning the interpretation of electro-titrimetric methods of analysis. G. Duyckaerts (*Ind. Chim. Belge*, 1953, **18** [8], 795–808).—The mathematical representation of polarisation curves obtained by different workers is discussed, and the effect of diffusion phenomena and the kinetics of electrode reaction is noted. Methods of potentiometric and amperometric titration are discussed on the basis of polarisation curves.   H. H. M. JONES

12. High frequency titration. O. L. Kaptsan and V. A. Teplyakov (*J. Anal. Chem.*, *U.S.S.R.*, 1953, **8**, 131–139).—A review with 35 references.
        G. S. SMITH

13. Determination of indicator error by the method of I. M. Korenman. I. L. Teodorovich (*J. Anal. Chem.*, *U.S.S.R.*, 1953, **8**, 184).—A slight modification of Korenman's formula (*Brit. Abstr. A* I, 1937, 196) is put forward.   G. S. SMITH

14. Indirect analysis of a three-component system. H. Bode (*Z. anal. Chem.*, 1953, **139** [2], 101–107).—Details, including the method of calculation for the analysis of a three-component system are given.
        G. P. COOK

15. Analytical use of the reaction of hydrolysis of potassium manganate. S. F. Zavgorodny (*J. Anal. Chem.*, *U.S.S.R.*, 1953, **8**, 172–174).—The determination of manganate by hydrolysis to MnO_2 by means of CO_2, $KHSO_4$, etc., gives low results except in the presence of at least 10 mol. of $KMnO_4$ to each mol. of K_2MnO_4.   G. S. SMITH

2.—INORGANIC ANALYSIS

16. Determination of small amounts of water-reactive hydrides in presence of inert salts containing base. M. D. Banus and J. B. Vetrano (*Anal. Chem.*, 1953, **25** [8], 1268–1269).—An apparatus and technique are described for determining water-reactive hydrides, in the presence of inert salts containing base, with an average precision of 2 per cent. for a concentration range of 1 to 5 millimoles of hydride per 100-g sample. The volume of hydrogen evolved when these hydrides react with water or dilute aqueous acid in a closed system is measured by water displacement.   O. M. JONES

17. Determination of alkalies in coal and coal ash. D. Logie and J. E. Rayner (*J. Inst. Fuel*, 1953, **26**, 146–150).—The Lawrence Smith method gives low and erratic results unless an ignition temp. of 1000° C is used to give complete extraction of coal ash. A wet oxidation method is described. involving perchloric and nitric acids, for the extraction of alkali metals direct from coal. The Na is determined as Na Zn uranyl acetate and the K as perchlorate; alternatively both metals can be determined by spectrography or other physical method.   T. H. BLAKELEY

18. Determination of potassium and traces of sodium in some potassium salts. C. Jackson (*Analyst*, 1953, **78**, 599–602).—For determination of

Na in certain K salts of weak acids, the sample is titrated with $0\cdot2$ N HClO$_4$ in glacial acetic acid. A small controlled excess of HClO$_4$ is added. The KClO$_4$ is removed by filtration and, after evaporation of the filtrate to dryness, Na is determined as sodium zinc uranyl acetate. It is thus possible to determine, volumetrically, the total equiv. alkali and, gravimetrically, both Na and K in the same sample. In the absence of obvious interfering ions (e.g., NH$_4$·, Li·, PO$_4$‴) the method is suitable for Na and K estimation in a variety of salts.

A. O. JONES

19. **Use of dilituric acid in the determination of potassium.** R. E. Press and K. A. Murray (*J. S. Afr. Chem. Inst.*, 1953, **6**, 17–21).—The reagent gives a ppt. with $0\cdot1$ mg of K, but for a determination, 10 to 35 mg of K are desirable. Five ml of solution are added to 20 ml of $0\cdot05$ N dilituric acid. The solution and ppt. are mixed occasionally and filtered through a Jena No. 4-grade sintered glass Gooch adapter. The beaker and ppt. are washed with $10 \pm 0\cdot5$ ml of freshly prepared saturated K diliturate solution. Two ml of N CuSO$_4$ solution are added to the filtrate with constant stirring. The ppt. and solution are mixed occasionally and filtered as previously, and washed with $\simeq 10$ ml of water. The filtrate is neutralised with aq. NH$_3$ and the slight ppt. of Cu(OH)$_2$ is dispelled with a few drops of acetic acid. The Cu is titrated iodimetrically and the K is equivalent to twice the difference between the titre and a concurrently determined distilled water blank. Na and Li and anions not interfering in the Cu titration do not affect the results; in fact, the presence of a little Na is desirable.

J. DAVIS

20. **Volumetric determination of potassium after precipitation as potassium tetraphenyl boron.** H. Flaschka, A. M. Amin and A. Holasek (*Z. anal. Chem.*, 1953, **138** [4], 241–244).—The test solution is acidified with a few drops of 1 per cent. acetic acid and warmed from 70° to 80° C. At least twice the theoretically required amount of a 3 per cent. aqueous solution of Na tetraphenylborate is added with stirring and subsequently one or two drops of $0\cdot02$ N AlCl$_3$. The mixture is allowed to cool to room temp. with continued stirring, and then filtered through a filter stick. The ppt. is washed with a few drops of water, with 1 to 2 ml of washing solution and then with 2 to 3 drops of water. The ppt. is dissolved from the plate of the filter stick and the walls of the vessel with 2 to 4 ml of acetone, and $0\cdot5$ to 1 ml of acetone is blown from the inside to the outside of the filter stick. One or two ml (according to the amount of ppt.) of saturated HgCl$_2$ solution, a measured (excess) vol. of $0\cdot01$ N NaOH and a little $0\cdot02$ per cent. alcoholic solution of methyl red are added and the mixture is boiled for a short time. A 20 per cent. solution of KI is added dropwise until the HgI$_2$ at first pptd. is dissolved. The mixture is titrated with $0\cdot01$ N HCl to a strong red, boiled to expel CO$_2$ and back-titrated with the NaOH. When K < 100 μg, a blank is carried out. The washing solution is prepared by dissolving K tetraphenylborate in acetone (15·0 mg dry solid in 1 ml acetone) and adding $0\cdot4$ ml of this solution to 100 ml of water. For quantities of K $\simeq 34\cdot0$ μg the deviations are $\pm 2\mu$g, and for 3000 μg ± 20 μg. W. GOOD

21. **Separation of caesium chloride by sublimation from mixtures with potassium and rubidium chlorides.** W. D. Treadwell and W. Werner (*Helv. Chim. Acta*, 1953, **36** [6], 1445–1454).—Comparative experiments

on the sublimation of CsCl, RbCl and KCl are described. The separation of CsCl by vacuum sublimation from equimolecular mixtures of salts is investigated. With N as carrier gas, the sublimates obtained at the lowest possible pressures ($\simeq 10^{-5}$ mm of Hg) form discrete crystallites of characteristic habit. Detection of the alkali ions present is described and microphotographs are shown. Complete separation of CsCl from KCl is attained at 430° C for mixtures where > 45 moles per cent. of CsCl are present. At 440° C separation is complete for a narrower range of mixtures, whilst at 480° C no complete separation is recorded. RbCl is not completely separable from CsCl at either 430° or 440° C, the best separations being $\simeq 70$ mol. per cent. CsCl in the sublimate to 50 mol. per cent. CsCl in the solid phase.

D. R. GLASSON

22. **Concentration method in determination of traces of copper in salts of iron.** V. T. Tchuiko and V. J. Lotareva (*Ukr. J. Chem.*, 1951, **16**, 612–615).—In the presence of Cu·· as microcomponent and Fe·· as macrocomponent, concentration can be carried out by means of partial precipitation of iron in the form of sulphide.

Determination of copper with sodium diethyl-dithiocarbamate by measuring the yellow-brown colour of the copper complex is described. The metals interfering are Ni, Co, and Bi, all of which form intense colorations with the reagent, while Cd, Hg, Ag and Pb, were found to give slight colorations. To minimise the interference of these metals, Cu, which was extracted with CCl$_4$, was transferred into the water layer by means of KCN, and the cyanide complex was destroyed by means of KBrO$_3$; Cu was then determined colorimetrically. Quantities of 5 to 10 μg Cu·· in presence of 10 μg Fe··, 20 μg Bi···, 5 μg Cd·· and 10 μg Ni·· have been determined in one sample. If quantities of Fe··· are present, Cu is separated in form of the carbamate, and extracted with CCl$_4$ from ammoniacal citric acid solution and with KCN from Bi and other elements not forming complexes. To separate Cu from Co and Ni, dimethylglyoxime was introduced before the extraction. Co forms a complex that is unaffected by carbon tetrachloride, whereas the Ni ppt. can be filtered off. Extracted iron was removed by oxidation with Br water; up to 2·5 per cent. of Fe was permissible.

To determine completion of passage of Cu into the water layer, the CCl$_4$ layer is separated and some sodium diethyldithiocarbamate is added to the water solution; the CCl$_4$ extraction is then repeated. Also Cu is transferred into water solution with KCN. To the combined water extracts a small excess of HCl is added and the solution is evaporated almost to dryness after addition of KBrO$_3$. The remainder is then dissolved in water and added to a solution of sodium diethyldithiocarbamate and alkali; the coloration is compared with a standard. The method is accurate in presence of Cu up to 4×10^{-4} per cent.

E. PREMUZIC

23. **p-Anisidine—a sensitive reagent for cupric ion.** A. Kh. Batalin (*J. Anal. Chem., U.S.S.R.*, 1953, **8**, 182–183).—Aq. solutions of *p*-anisidine and Cu·· salts give a yellow green ppt. changing to deep violet in 5 to 8 min. A violet colour without ppt. occurs with small amounts of Cu with a detection limit of 4 μg in 1 ml. In presence of a large excess of CNS′ or I′ higher concn. of Cu give a red ppt. and small concn. a cherry-red solution. Decoloration is effected with aq. NH$_3$, citrates, pyrophosphates, and

a large excess of mineral acid. Two to three drops of a reagent, prepared by dissolving 1 ml of a saturated solution of p-anisidine in 100 ml of 0·1 N CNS' or I', added to 1 ml of Cu solution, 0·6 μg can be detected. The sensitivity is unaffected by 500 times the amount of Mn, Al, Zn, Mg, Cd, Ni, Cr, alkali metals, alkaline earths, and Fe¨, by 50 times the amount of Co, Ag, Sn, U, Ce, Hg¨, and by 5 times the amount of Sb and Hg˙. NaF can be added to prevent interference from Fe˙˙˙.   G. S. SMITH

24. Simultaneous colorimetric determination of copper, cobalt, and nickel as diethyldithiocarbamates. J. M. Chilton (*Anal. Chem.*, 1953, **25** [8], 1274–1275). —In the method described for the simultaneous colorimetric determination of copper, cobalt and nickel (particularly of microgram quantities in the presence of substantial amounts of other metals), their diethyldithiocarbamates are formed at a pH of 8·5 to 9·0 in an aqueous solution to which pyrophosphate and citrate have been added; the coloured complexes are subsequently extracted with carbon tetrachloride. Absorption spectra of the three diethyldithiocarbamates solutions in carbon tetrachloride are presented.   O. M. JONES

25. Extraction of cations as salts of fatty acids. I. Beryllium as butyrate. A. K. Sundaram and S. Banerjee (*Anal. Chim. Acta*, 1953, **8** [6], 526–529). —Metallic ions can be extracted by organic solvents in the presence of the higher fatter acids. A range of organic solvents is investigated as extractants for Be in the presence of butyric acid. Chloroform gave optimum extraction of 94 per cent. with 10 to 15 ml of butyric acid in 50 ml of solvent adjusted to pH 9·3 by the addition of NH_3. Four extractions remove 99 per cent. of the Be. A salt effect is also studied.   W. C. WAKE

26. Removal of manganese prior to calcium and magnesium precipitations. B. L. Ingram and L. Bean (*Anal. Chem.*, 1953, **25** [8], 1217–1219).— Proposed modifications of the suggested A.S.T.M. $KMnO_4$ method for removal of Mn from cement prior to Ca and Mg pptn. are described. The modifications include the pptn. of the hydrated MnO_2 in a solution ammoniacal to methyl red, substitution of digestion at 80°C for boiling, washing of the pptd. Mn oxides with hot dil. HCl followed by re-pptn. of the Mn and elimination of the dil. HNO_3 washing. Results for the $KMnO_4$ procedure show that it is far superior to the present ammoniacal bromine solution method.   G. P. COOK

27. The polarographic behaviour of some elements in concentrated calcium chloride solution. I. General introduction. Certain problems arising from the use of 5 M calcium chloride. G. F. Reynolds, H. I. Shalgosky and Y. J. Webber (*Anal. Chim. Acta*, 1953, **8** [6], 558–563).—The polarographic behaviour of a base electrolyte of $5M$ $CaCl_2$ is studied with a view to its use for estimating trace metals in Ca; pH measurements are unstable and need to be inferred from those obtained on ten-fold dilutions. Solutions of Cu, Cr, Ni, Pb, Cd, and Mn in 5 M $CaCl_2$ were used with a single sweep cathode-ray polarograph (see J. E. B. Randles, *Trans. Faraday Soc.*, 1948, **44**, 322–338). The need to correct the Ilkovič equation for the viscosity of the medium is shown by agreement of the corrected figures with the diffusion currents measured on a Cambridge photographic polarograph, but those measured with the cathode-ray polarograph are still low. Possible reasons for this are discussed.   W. C. WAKE

28. The polarographic behaviour of some elements in concentrated calcium chloride solution. II. The polarography of chromium, nickel and lead. G. F. Reynolds, H. I. Shalgosky and Y. J. Webber (*Anal. Chim. Acta*, 1953, **8** [6], 564–571).—The reduction $Cr^{III} \rightarrow Cr^{II}$ is convenient for $5M$ $CaCl_2$ solution especially as a stable form of Cr^{III} can be obtained at pH $> 3·5$ after heating to 100° C. For nickel only one reduction is possible and the half-wave potential of this, calculated as $+ 0·31$ V, is found at $+ 0·32$ V, the over-voltage usually associated with hydration being absent in 5 M $CaCl_2$. The half-wave potential found for Pb is $+ 0·29$ V. The diffusion current increases slightly with rise in pH and re-calibration is necessary for extremes of the pH. The reduction steps of Cr, Ni, and Pb overlap, but the wave heights are additive. At a pH of approx. 7 only Pb remains in solution and can be determined, but no reliable method for Cr and Ni in one solution could be devised.   W. C. WAKE

29. A precise heterometric determination of calcium in the presence of large excesses of magnesium. M. Bobtelsky and I. Bar-Gadda (*Anal. Chim. Acta*, 1953, **9** [2], 168–170).—(cf. *Anal. Abstr.*, 1954, **1**, 51). Assuming the salts are soluble in 90 per cent. C_2H_5OH, the Ca is titrated with neutral K phthalate to the max. of the optical density with an accuracy of better than 1 per cent. in the presence of a ten-fold excess of Mg.   W. C. WAKE

30. Determination of small amounts of calcium in magnesium-free brine. P. J. Hol and G. H. Leferink (*Chem. Weekbl.*, 1953, **49**, 733–735).—In titrating the Ca in pickling brine (500 ml of brine and 400 ml of boiling water gives a mixture at 50° $\pm$ 3° C) with di-sodium ethylenediaminetetra-acetate, in presence of a small amount of Mg, and with Eriochrome Black-T as indicator, colour changes are best observed by placing the titration beaker on a plate of milk-white glass illuminated from below. The Mg is added preferably in a buffer solution (20 ml per titration) containing 54 g NH_4Cl, 350 ml aq. NH_3 (25 per cent. NH_3) and 2 g of a Mg complex (supplied by B. Siegfried, Zofingen) per litre. Titrations are accurate to 0·02 mg of Ca per litre.   P. S. ARUP

31. Radium determination by alpha counting. H. W. Kirby (*Anal. Chem.*, 1953, **25** [8], 1238–1241). —A method is described which reduces the amount of labour and equipment involved in the assay of carrier-free radium. A dilute hydrochloric acid solution of the radium sample is passed through a short column of copper powder to remove polonium. The effluent is mounted on glass discs and alpha-counted 4 to 5 hr. after mounting. Twenty-four hours after mounting, the samples are counted again and the percentage increase in counts is used to determine a correction factor for the growth of radon and its daughters. The method is accurate and precise to better than 1 per cent. when the counting rate is sufficiently high to make the counts statistically reliable. The precision of the method is satisfactory with 44 counts/min. (4×10^{-11} g of radium).   O. M. JONES

32. Rapid titrimetric determination of zinc in aluminium and aluminium alloys. F. E. Faller (*Z. anal. Chem.*, 1953, **139** [1], 14–19).—For the analysis of pure Al and Al alloys containing 0·05 to 0·2 per cent. Zn, 5 g of the sample is dissolved in 50 ml of 25 per cent. NaOH and 15 ml of saturated hydrazine sulphate solution are added. After boiling, the solution is filtered, and the residue is washed

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with hot water. The filtrate is heated to boiling and treated with 10 ml of 20 per cent. Na_2S soln., the ppt. formed being filtered and washed with 0·1 per cent. Na_2S soln. and then dissolved in 33 per cent. HCl. This soln. is boiled until all H_2S is removed, then cooled, and 2 ml of 50 per cent. citric acid (to mask small quantities of Al) and 10 ml NH_4OH are added. The pH is adjusted to 8·5 to 9·5, and solid indicator is added (1 part Eriochrome Black to 500 parts NaCl) until a wine-red colour appears. The solution is titrated with 0·005 M di-sodium ethylenediaminetetra-acetic acid to a blue end-point. One ml of 0·005 M reagent ≡ 0·3269 mg Zn. The accuracy is ± 0·002 to 0·005 per cent. For the analysis of Al alloys containing 0·1 to 8·0 per cent. Zn, the titrant is 0·025 M, and 1 g of sample is taken. Accuracy is ± 0·01 to 0·05 per cent. Cu, Fe and Mn interfere slightly.   G. P. Cook

33. Determination of zinc by means of sodium diethyldithiocarbamate. J. E. O. Mayne and G. H. Noordhof (*Analyst*, 1953, **78**, 625).—The turbidi-metric method of Atkins (*Brit. Abstr. B*, 1935, 752) for determination of Zn in water has been adapted for use with the Spekker absorptiometer. Gum arabic proved unsatisfactory as protective colloid, but satisfactory results were obtained with polyvinyl alcohol (prep. from commercial polyvinyl acetate described). The relation between the turbidity determined on the absorptiometer and the concn. of Zn is linear over a definite pH range. The calibration is affected by the anions present and the method is available only when the composition of the solution is known or when it is possible to make the calibration under the same conditions as determination.   A. O. Jones

34. An alkalimetric method for the analysis of solutions containing zinc hydroxide and sodium hydroxide. M. M. Tiller (*J. Indian Chem. Soc., Ind. News Ed.*, 1952, **15**, 207–208).—NaOH and $Zn(OH)_2$ are estimated in the presence of each other by (*a*) estimating the total alkali in solution by the addition of an excess of KI and titrating with 0·1 N HCl, and (*b*) precipitating the Zn held as a K Zn iodide complex as the basic carbonate and estimat-ing by KI or $Na_2S_2O_3$.   D. E. Blenford

35. Interaction of cadmium and zinc salts with sodium hydroxide, carbonate, and orthophosphate. J. C. Clayton (*Dissert. Abstr.*, 1953, **13** [2], 173).—The reactions were studied amperometrically, conductimetrically and potentiometrically. With sulphate as the anion and NaOH as titrant, end-points occurred when less than the theoretical amount of base had been added. For nitrates and NaOH the end-points occurred at 100 per cent. equivalence. The influence of the anions in the basic salt was studied amperometrically. With Na_2CO_3, stoicheiometric end-points were attained except for $ZnSO_4$ for which no reproducible results were possible. Slight differences between potentio-metric curves and those from either amperometric or conductimetric techniques were observed with orthophosphate. The last two of these methods gave end-points at the 100 per cent. equivalent of titrant.   C. D. Lafferty

36. The use of ion exchangers for the separa-tion, concentration and determination of traces of cadmium in uranium. Z. Dizdar (*Rec. Trav. Inst. Rech. Structure Mat.*, 1953, **2**, 85–88).—Cadmium in uranium is determined by adsorption on a column of amberlite IR-120 resin. The uranium is eluted

with 0·5 N oxalic acid and the cadmium with N HCl. The cadmium is determined polarographic-ally.   W. Manz

37. The use of sodium fluoride as carrier in the spectrographic determination of cadmium and boron in uranium. Natalija Belegisanin (*Rec. Trav. Inst. Rech. Structure Mat.*, 1953, **2**, 27–30).—The Scribner - Mullin method for the spectrographic determination of impurities in uranium by the carrier distillation process has been modified by using NaF as the carrier material (10 to 15 g for 100 mg of U_3O_8). The smallest quantity of Cd determined is 3×10^{-8} g in 100 mg U_3O_8 with an accuracy of ± 15 to 20 per cent. by a Hilger E478 spectrograph, Ilford ordinary plates, 25 sec. exposure, Foster electrodes 4 mm apart and arc from 70 V and 6 amp. The intensity of the ultimate line of Cd 2288·01 A is measured with Be as internal standard. Boron is estimated by its characteristic doublet down to 1×10^{-8} g in 100 mg U_3O_8 with same accuracy as for Cd with Be (4×10^{-4} g added per 100 mg U_3O_8) as internal standard but with carbon elec-trodes.   W. Manz

38. A contribution to the spectrochemical analysis of boron. Marija Mitrović (*Rec. Trav. Inst. Rech. Structure Mat.*, 1953, **2**, 23–26).—Boron in aq. boric acid, borosilicate glass (Pyrex), silica, graphite, or Lendava naphtha-ashes is estimated from its spectral lines $\lambda = 2496·778$ and $\lambda = 2497·773$ on a Littrow spectrograph by con-densed spark and from d.c. arc discharge spectra. Better results are obtained from d.c. arc (220 V, 8 amp.) by means of 5-mm spectrographically pure carbon electrodes having a crater 10 mm deep and 2 mm diameter provided in the lower electrode; a gap of 2 mm is maintained between the electrodes. Quantities of boron down to 3×10^{-8} g can be detected and 1×10^{-7} g boron can be determined.   W. Manz

39. Colorimetric micro determination of boron by the curcumin – acetone solution method. L. Silver-man and K. Trego (*Anal. Chem.*, 1953, **25** [8], 1264–1267).—A study is described of the effect of varia-tions in the amounts of reagents used in the boron - curcumin reaction, as applied to the colorimetric determination of boric acid or borate in solutions of low solid content. The procedure is effective in water solution for determining detectable amounts of B (0·02 μg, contained in a 10-ml standard volume of solution plus reagents). Precautions are described to prevent contamination of reagents and apparatus at low B contents (0·1 μg or less). Precision and reproducibility of the results in ranges between 0 and 100 μg are given, and this colorimetric method is compared for different concentrations of boric acid with the potentiometric method. Application is limited in this paper to solutions containing little iron, aluminium, etc., but requiring precise analytical methods for B in the range of 0·2 to 25 μg.
                        O. M. Jones

40. Determination of micro-quantities of boron with curcumin. B. V. Zaletel (*Rec. Trav. Inst. Rech. Structure Mat.*, 1953, **2**, 31–59).—Three new procedures for the determination of boron in the range 0·03 to 1·00 μg with an accuracy of ± 0·03 μg based on the curcumin salicylic acid complex are given. An intense scarlet-red colour of the complex in the presence of paraffin is reported; also reported is a new micro method based on photo-electric measurement of the blue colours appearing after addition of alkali to the curcumin salicylic acid

complex with B. Boron contents ranging from 1.5×10^{-9} to 5×10^{-8} g of B per ml with an error of 2 to 3 per cent. are estimated by this reaction, which is entirely specific for boron. The mechanism of the reaction is studied. A plot of curcumin added against colour intensity shows steps. The addition of more curcumin increases the colour intensity in steps rather like polarographic steps corresponding to ratios $1 : 6$, $1 : 10$, $1 : 12$ of molecules of boric acid to curcumin; these steps are independent of the conductivity and pH of the solution. A minimum of 0.000645 g of curcumin is needed for $3 \mu g$ of boron. Increase in salicylic acid concentration leads to increase in colour intensity, which reaches a maximum and then decreases. Increase in amount of HCl gives a decrease in colour intensity. For $3 \mu g$ of boron, 0.000516 g curcumin, 0.0016 to 0.0018 g salicylic and 0.03 ml $(2 \times 10^{-4}$ moles) of HCl $(2 + 1)$ represent optimal quantities.

W. Manz

41. A spot test for aluminium. A. Lewandowski (*Roczn. Chem.*, 1953, **27** [2], 209–215).—In order to detect Al a few drops of the examined solution are placed on a piece of filter-paper, which is then immersed for 5 min. in M aq. NH_3 solution containing 10 per cent. of NH_4NO_3. After the paper has been washed in water and dried superficially, a mixture of NaF or KF and an indicator is sprayed on it, so colouring the spots containing Al. A list of sensitive indicators some of which are capable of detecting 10 p.p.m. of Al is given. Elimination of interfering ions is discussed; Ti, Zr, Mn, Be, Ta and Nb interfere most.   S. K. Lachowicz

42. Determination of complexes formed by trilons with the rare earths. F. Boulanger (*Chim. Anal.*, 1953, **35** [10], 253–254).—Trilon A (nitrilotriacetic acid) and TrilonB (ethylenediaminetetra-acetic acid) give stable complexes with most metals. Simple experiments indicate that the complex ions formed with Pr are $[Pr (A)_2]'''$ and $[Pr (B)]'$. Progressive acidification from pH 6 of a solution containing the complexed rare earths and an oxalate leads to the pptn. of the oxalates of La at about pH 6, then Nd, and Pr; at about pH 5, Sa; pH 4.5, Gd; and pH 4, Er. At pH < 4 the Yt group of earths are precipitated. Cu^{II} in acid solution displaces the rare metals from their complexes.   W. C. Wake

43. Radiometric determination of small amounts of thallium. T. Ishimori (*Bull. Chem. Soc. Japan*, 1953, **26** [6], 336–338).—Tl is precipitated with hexammine cobaltic trichloride that has been labelled with ^{60}Co. The radioactivity of the ppt. is used as a measure of the Tl in the pptd. $Co(NH_3)_6TlCl_6$ provided that radioactive Tl is absent.   C. D. Lafferty

44. Qualitative test for scandium employing 2:5-dihydroxy-1:4-benzoquinone. L. Pokras and M. Kilpatrick (*Anal. Chem.*, 1953, **25** [8], 1270–1272).—A solution of 2:5-dihydroxy-1:4-benzoquinone in acidified alcohol produces a pinkish precipitate with 0.04 mg or more of Sc. The effects of various ions and salts are described. Fluoride appears to increase the sensitivity of the test.   O. M. Jones

45. The use of barbituric acid for the photometric determination of cyanide and thiocyanate. E. Asmus and H. Garschagen (*Z. anal. Chem.*, 1953, **138** [6], 414–422).—CN′ and CNS′ can be determined photometrically by use of barbituric acid, Cl being added in the form of chloramine. By using solutions whose pH values lie between 2 and

10, CN′ can be estimated from 1 μg to 1 mg per litre and CNS′ from 1 μg to 2.5 mg per litre. In the CN′ determination, 1 ml of 1 per cent. aq. chloramine solution is added to 25 ml of the sample, and after shaking, the solution is covered and set aside for 1 min. Three ml of barbituric acid reagent are added with shaking, and the solution is set aside for 8 min. before the photometric determination. In the CNS′ determination, 1 ml of 0.1 per cent. $FeCl_3.6H_2O$ solution and 1 ml of 1 per cent. chloramine solution are added to 25 ml of the sample. After mixing, the solution is allowed to stand 2 min. and 3 ml of the barbituric acid reagent are added with shaking. Five to eight min. later the photometric determination is made against a blank. In both cases, the optical measurement must be completed within 15 min. of the addition of the reagent. The reagent is prepared by dissolving 3 g of barbituric acid in 15 ml of pyridine, diluting with H_2O, adding 3 ml of conc. HCl and making up to 50 ml. A Hg-lamp is used in the photometer, the solution having a max. absorption at 578 mμ.   J. H. Waton

46. Determination of silicon by precipitation of the silicomolybdic yellow complex by quinoline. M. Armand and J. Berthoux (*Anal. Chim. Acta*, 1953, **8** [6], 510–525).—A study of the conditions and of the products leads to a method for the gravimetric determination of silicon by pptn. of the complex $SiO_2.12 MoO_3.4(C_9H_7N).2H_2O$. This ppt. is dried for one hour at 150° C.   W. C. Wake

47. Spectrophotometric determination of tin (IV). H. Teicher and Louis Gordon (*Anal. Chem.*, 1953, **25** [8], 1182–1185).—The sample containing from 50 to 7000 μg of Sn^{IV} is evaporated to fuming with 5 ml of conc. H_2SO_4. The solution is cooled and diluted to 100 ml, duplicate 10-ml aliquots are taken and 5 ml of 0.1 per cent. haematoxylin (in 95 per cent. alcohol) and $0.005 N$ in HCl) are added. Dilution to 50 ml follows and the absorbancy is measured at 515 mμ after 1 hour. The Sn^{IV} content is found by reference to a suitable calibration graph. Beer's law is not completely followed but the curves are reproducible. The method is sensitive to pH change, therefore conditions must be rigorously standardised. The error is $\simeq \pm 1$ per cent. Interfering elements are Fe^{III}, Sb^{III}, Bi^{III}, Ti^{IV}.   G. P. Cook

48. Determination of tin by distillation of the iodide. Phosphor and antimony bronzes. J. Besson (*Chim. Anal.*, 1953, **35** [10], 241–243).—The previous method (*Brit. Abstr. C*, 1952, 535) of determination by distillation of SnI_4 applied to siliceous bronzes; the present study shows that it cannot be recommended for antimony bronzes unless there is strict control of heating temperatures. In these the mixed oxides are distilled at 500° C with NH_4I after calcination at 850° C. Thermobalance graphs show the limited temperature range available around 850° C. The method is not suitable for phosphor bronzes.   W. C. Wake

49. Reversion method for absorptiometric determination of traces of lead with dithizone. H. M. Irving and E. J. Butler (*Analyst*, 1953, **78**, 571–578).—The method described for determination of Pb in small samples of biological materials involves wet ashing of the sample, extraction of all the Pb (and any Bi present) with dithizone, separation of most of the Bi, and absorptiometric determination of the Pb at 620 mμ by a reversion technique. The molecular absorbancy of the dithizone extract is the

sum of the mol. absorbancies of Pb dithizonate (PbDz$_2$), excess dithizone (HDz) and coloured impurities. If Pb is now removed and thereby replaced by the equivalent amount of HDz, the increase of absorbancy $\propto$ the concn. of Pb. Up to 2 mg of Pb can be determined in presence of up to 20 μg of Bi and at least 100 μg of Cu. A. O. JONES

50. Polarographic method for the determination of lead in iron ores and products. C. Gandon (*Chim. Anal.*, 1953, **35** [10], 251–260).—The solution in HCl is reduced by NH$_2$OH.HCl, adjusted to pH 3 by NH$_3$ and the Cu content determined in an aliquot portion. To the remainder, sufficient Cu^{II} is added so that the ratio Cu/Fe is > 0.005, and KCNS is added. Ten ml of the solution is then polarographed and a further 10 ml is polarographed after the addition of 1 ml of a solution containing a known quantity of Pb. Results are quoted for ferro-manganese and pyrites. W. C. WAKE

51. A precise heterometric micro-determination of lead or citric acid. M. Bobtelsky and B. Graus (*Anal. Chim. Acta*, 1953, **9** [2], 163–167).—A titration resulting in the formation of a suspension is followed by means of a photo-electric measurement of optical density. The addition of citrate to Pb in aqueous solution gives steadily increasing optical density (D) until a max. is reached. Further addition produces no increase at first, but after a small excess of citrate D falls owing to the formation of a soluble complex. The reverse titration is slightly more complex and extrapolation is required to determine citric acid. Titration in 50 per cent. aqueous C$_2$H$_5$OH gives greater accuracy. W. C. WAKE

52. Use of the polarograph in the determination of lead in gasoline engine fuel, lubricants and deposits. B. W. Swanson and P. H. Daniels (*J. Inst. Petrol.*, 1953, **39**, 487–497).—The Pb is extracted by means of the Schwartz reagent (KClO$_3$ - NaCl in HNO$_3$) and then, after evaporation of the acid solution, determined (*a*) polarographically in presence of NH$_4$ acetate and magenta or (*b*) amperometrically by titrating with 0.01 MK$_2$Cr$_2$O$_7$ in presence of KNO$_3$. By these methods increased accuracy and reduced time of operation are secured. With gasoline agreement is shown with standard methods within the specified limits of $\pm$ 0.04 ml per imp. gal. Procedures are described for lubricating oils, engine sludges, and deposits. J. G. KING

53. Dithizone as indicator in titrations by precipitation and by complex formation. Determination of lead and cadmium. E. Bovalini and A. Casini (*Ann. Chim., Roma*, 1953, **43**, 287–292).—A CCl$_4$ solution of dithizone is used as an indicator in determinations of Pb and Cd by titration with disodium ethylenediaminetetra-acetate, and (with poorer results) in titrations of Cd with K$_4$Fe(CN)$_6$. J. K. LANDQUIST

54. Colorimetric determination of titanium with sulphosalicylic acid. M. Ziegler and O. Glemser (*Z. anal. Chem.*, 1953, **139** [2], 92–96).—The sample, in $\simeq$ 0.1 N H$_2$SO$_4$, is added to 5 ml of 20 per cent. sulphosalicylic acid, one drop of 5 per cent. FeCl$_3$ is added, and the solution is titrated with 10 per cent. aq. NH$_3$ until the colour changes from red to yellow. Twenty ml of buffer soln. (6.44 g of Na acetate and 10.45 ml of 96 per cent. acetic acid diluted to 1 litre with water) are added, followed by 5 ml of 4 per cent. thioglycollic acid after shaking. The absorbancy is measured after 10 min. at 445 mμ.

The reproducibility of the method is good, even in presence of Mg, Cu, Al, Fe, Mn, Ca, Va and Zn. G. P. COOK

55. Determination of impurities in titanium metal. J. M. Thompson (*Anal. Chem.*, 1953, **25** [8], 1231–1235).—Methods are described for determining impurities in titanium metal sponge. Iron is determined by the coloured thiocyanate - iron complex; a modification of the method for cast and wrought forms of Ti is described. In a modified semi-micro Kjeldahl method for nitrogen, a simple sulphuric acid digestion converts the nitrogen in the sponge to ammonium sulphate, but in cast and wrought Ti, hydrogen peroxide treatment is required. Magnesium is determined by either the phosphate method or oxine method, and manganese by oxidation to permanganic acid with potassium periodate and comparison of the colour developed with standards. Chloride is determined gravimetrically by dissolving in HF, adding boric acid and then precipitating the chloride with silver nitrate. Volhard's volumetric method has been found to be satisfactory for chloride contents above 0.10 per cent. Reproducibilities and accuracies of the methods are indicated. O. M. JONES

56. Preparation and properties of 2-(2-hydroxy-3:6-disulpho-1-naphthylazo)benzenearsonic acid (Thorin). Analytical reagent for thorium. D. W. Margerum, C. H. Byrd, S. A. Reed and C. V. Banks (*Anal. Chem.*, 1953, **25** [8], 1219–1221).—The Thorin (also known as Thoron or Naphtharson) is synthesised by the coupling of 2-nitroaniline with Na$_2$HAsO$_3$ by the Bart reaction to form 2-nitro benzenearsonic acid, which is reduced to 2-amino benzenearsonic acid (**I**) by FeCl$_2$. Compound (**I**) is then coupled with the di-sodium salt of 2-naphthol-3:6-disulphonic acid by the Griess reaction. The yield is 33 per cent. and the product (the tri-sodium salt) is > 99 per cent. pure. The acid dissociation constants for Thorin are pK$_3$ = 3.7 and pK$_4$ = 8.3 for the arsono-hydrogens and pK$_5$ = 11.8 for the naphtholic hydrogen. G. P. COOK

57. Quantitative insolubility of thorium oxalate. H. I. Kall and Louis Gordon (*Anal. Chem.*, 1953, **25** [8], 1256–1258).—An investigation is described into the solubility losses of thorium oxalate in dilute acid media. The effects of pH, variation in oxalate and Th concentrations, and digestion time are reported. The results indicate that the oxalate method should be avoided whenever possible for determining Th, but if used, *e.g.*, to separate Th from Ti, Zr, or phosphate, NH$_4$ salts should be absent or else rare earths should be present as carriers. Hexamine can be used to precipitate Th quantitatively in the presence of ammonium salts or the rare earths, but Ti, Zr, or phosphate interfere. O. M. JONES

58. Determination of nitrate in sodium nitrite. C. L. Johnson (*Anal. Chem.*, 1953, **25** [8], 1276–1277). —In the method described for determining nitrate in nitrite solutions, an aqueous solution of the sample is added to cold concentrated sulphuric acid in an evacuated bottle. The nitrite is converted quantitatively to nitrososulphuric acid. The nitrate is then titrated with ferrous sulphate without interference from nitrite or nitrososulphuric acid. Results tabulated indicate an accuracy within $\pm$ 0.10 per cent. in the range 0 to 10 per cent., within $\pm$ 0.20 per cent. in the range 10 to 50 per cent., and within $\pm$ 0.4 per cent. at 90 per cent. potassium nitrate. O. M. JONES

59. New reagent for the detection of nitrite ion.
A. L. Misra, R. C. Mehrota and J. D. Tewari (*Z. anal. Chem.*, 1953, **139** [2], 89–92).—A dil. solution of 2-amino-4-chloromethylthiazole hydrochloride gives a red colour or ppt. with a neutral nitrite solution. Detection is possible in presence of NO_3', which only give a white cryst. ppt. The reagent is soluble in water and the reaction with NO_2' proceeds visually. The limit of detection is $1\,\mu g$. The colours observed for various concn. of reagent are included. A colorimetric method for the determination of NO_2' may be possible with this reagent.
G. P. COOK

60. Alkalimetric determination of phosphoric acid and phosphates. E. Rancke-Madsen and T. Kjaer-gård (*Acta Chem. Scand.*, 1953, **7** [5], 735–740).—A method for the determination of phosphate involving pptn. with $Ce(NO_3)_3$ is described. By use of a mixed indicator of 0·02 per cent. methyl orange and 0·1 per cent bromocresol green and by addition of a small quantity of NaCl, the solution is titrated to the first equivalence-point of phosphoric acid (H_2PO_4') with HCl. A small excess of aq. $Ce(NO_3)_3$ is added and the solution is titrated with standard alkali until the indicator assumes the same colour as at the first equivalence-point ($H_2PO_4' + Ce^{\cdots} \rightarrow CePO_4 + 2H^{\cdot}$). The results deviate from gravimetric determinations by 0 to 0·6 per cent. The theory of the method is discussed   A. R. MATHIESON

61. The analytical determination of trimetaphosphate in the presence of other phosphates
G. Ender (*Z. anal. Chem.*, 1953, **138** [6], 401–404).—The separation of metaphosphate from other phosphates is effected by pptn. with $Ba(ClO_4)_2$. The metaphosphate in the filtrate is then estimated by polarographic titration with bismuthyl perchlorate. Results for the metaphosphate determination in the presence of tripolyphosphate are good, but low in the presence of pyrophosphate and orthophosphate, although even then the values are of the right order.   J. H. WATON

62. Colorimetric determination of phosphoric acid with vanadate - molybdate. F. Scheffer and H. Pajenkamp (*Z. anal. Chem.*, 1953, **138** [4], 266–269).—A discussion of the accuracy of the colorimetric as opposed to the gravimetric method.
W. GOOD

63. Iodometric determination of hypophosphorous and phosphorous acids. Richard T. Jones and E. H. Swift (*Anal. Chem.*, 1953, **25** [8], 1272–1274).—Procedures described for determining hypophosphorous and phosphorous acids, alone or in mixtures, are based on the assumptions that (*i*) phosphorous acid can be oxidised quantitatively by excess of iodine in an essentially neutral solution in 1 hour, oxidation of hypophosphorous acid being negligible under the same conditions, and (*ii*) hypophosphorous acid can be oxidised quantitatively by excess of iodine in 1 to 2 N HCl in 3 hours. Results tabulated indicate that mixtures of these two acids can be analysed with a precision within 0·1 to 0·2 per cent. for concentrations of 0·02 formality or greater.   O. M. JONES

64. Separation of small amounts of arsenic, copper and bismuth from lead and zinc by means of diethylammonium diethyldithiocarbamate. N. Strafford, P. F. Wyatt and F. G. Kershaw (*Analyst*, 1953, **78**, 624–625).—In the scheme of analysis for trace metals previously described (Strafford *et al.*, *Brit.*

Abstr. C, 1945, 284), As, Cu and Bi are separated from Pb, Zn etc. by extraction with a solution of diethylammonium diethyldithiocarbamate (**I**) in $CHCl_3$ from the wet ash digest adjusted to at least 3 N in H_2SO_4 and 2N in HCl. Increase of the HCl concn. to 3 N is now recommended. It has been assumed that this separation is effective from solutions 2 N in HCl alone (Hart, *Brit. Abstr. C*, 1952, 218), but this is incorrect as almost all the Pb is extracted with Cu and Bi unless the HCl concn. is raised to 4 to 6 N. HCl is essential for success; even from 10 N H_2SO_4 as acid, $\simeq$ 85 per cent. of the Pb is extracted with the Cu and Bi. The reagent (**I**) is now available in the necessary degree of purity and a 1 per cent. solution is preferable to a reagent prepared *in situ* from CS_2 and diethylamine.
A. O. JONES

65. Determination of antimony combined with oxygen in minerals. P. de Vadder (*Chim. Anal.*, 1953, **35** [10], 248–250).—Flotation processes that are successfully used with Sb_2S_3 fail with Sb_2O_3, the proportion of which in minerals is of importance. Sb_2O_3 is soluble in acid K tartrate in 2 hours, whereas only 1·8 per cent. of Sb as Sb_2S_3 is dissolved. Dissolved Sb is estimated by titration with I after treatment with H_2S if As is present, or with $KBrO_3$ or $KMnO_4$ if it is absent.
W. C. WAKE

66. Comparative evaluation of qualitative reactions for bismuth based on the method of grinding. E. P. Ozhigov (*J. Anal. Chem., U.S.S.R.*, 1953, **8**, 175–177).—Various Bi minerals and ores were treated with H_2SO_4 and HNO_3, and the residues after evaporation of the acid were ground with pyrogallol, NH_4CNS, $Na_2S_2O_3.3H_2O$, or $(NH_4)_3Cr(CNS)_6$ in separate tests. The most sensitive reaction for Bi was obtained with NH_4CNS and the intensity of the yellow colour produced depended on the Bi content. Addition of Na_2SO_3 to the mixture prevented interference from Fe. 1·37 μg of Bi can be detected in a 0·03-g sample.
G. S. SMITH

67. Determination of vanadium (V) in oxalate solution and in presence of iron (III) and copper (II) by the dropping mercury electrode. G. de Angelis and N. Carugno (*Ric. Sci.*, 1953, **23** [9], 1593–1602).—V^{V} is determined polarographically in M K oxalate solution at pH 4·6 to 4·9 in presence of 0·01 per cent. gelatin soln. Results are best in absence of Cl' and in red light. Tables are given showing results obtained in presence of Fe^{III} and Cu^{II}. Quantities of V^{V} of 0·255 to 1·53 mg per 25 ml in presence of up to 100 mg of Fe^{III} and 1·54 mg of Cu are determined with an accuracy of $\pm$ 2 per cent.   BRITISH ABSTRACT

68. Polarographic determination of niobium in presence of tantalum with particular reference to niobites and tantalites. S. Vivarelli and D. Cozzi (*Chim. e. Ind.*, 1953, **35** [9], 637–638).—Nb is quantitatively and reversibly reduced on the polarograph at -0.395 V ($vs.$S.C.E.) $i_d/Cm^{2/3}\,t^{1/6}$ = 0·825 at 25° C from Nb^{V} to Nb^{IV} in the presence of Ta in 10N HCl containing 20 per cent. v/v of ethylene glycol. Fe^{III}, Mo^{V}, Cu^{II}, Ti^{IV} precede the Nb-wave, which is also interfered with by Pb^{II}, Sn^{II}, Sn^{IV}, As^{III}, Sb^{III}, V^{III}, W^{VI} and U^{VI}. A procedure for the chemical separation of Nb and Ta from the minerals is described. Tantalum is calculated by difference from the weight of the mixed oxides.   W. MANZ

69. Determination by radioactivity of mixed tantalum, niobium and titanium oxides. J. Beydon and C. Fisher (*Anal. Chim. Acta*, 1953, **8** [6], 538–545).—Two methods are presented. I. Irradiation with slow neutrons. The half-life periods of the isotopes of Nb and Ti which are formed are only 6·6 min. and 6 min., whilst Ta has an isotope with a half-life of 117 days. This isotope is conveniently determined from its gamma radiation after an interval of 24 hours. Ti is then determined colorimetrically. II. ^{95}Nb is obtained as a fission product of U and has a half-life of 35 days. A solution containing the three metals is treated with tannin after ^{95}Nb has been added. The pH is adjusted to 3·8 and tannin is added thus pptg. all Ta. The ppt. is filtered, washed, calcined and weighed. The filtrate is concentrated, adjusted to pH 6·0 and the remainder of the Nb is pptd. with tannin, the ppt. being treated as before. Weighed portions of both ppt. are taken for the Ti determination, the remainders having their radioactivity measured. If the amount of Ta is appreciable, part of the first tannin ppt. is fused with $KHSO_4$ to which Ta_2O_5 containing ^{182}Ta is added. This enables Ta to be evaluated. Nb activity is measured in both the tannin ppt. W. C. WAKE

70. Determination of oxygen in water in presence or absence of active reducing substances. K. Wickert and E. Ipach (*Z. anal. Chem.*, 1953, **139** [3], 181–188).—Dissolved O is determined by measurement of the colour produced with a tolidine – Br solution in the presence or absence of Mn˙˙ or Ce˙˙˙. In the presence of the oxidisable metal ions, total O and reducing substances are determined, whilst in their absence only reducing substances are estimated. From these data the Oxygen in the sample is calculated. C. G. TAYLOR

71. Chemistry of sulphur. XIII. The volumetric determination of pure alkali polysulphides. F. Fehér and H. J. Berthold (*Z. anal. Chem.*, 1953, **138** [4], 245–249).—A few tenths of a gram of the alkali polysulphide are dissolved and made up to a known volume with boiled water. To an aliquot part an excess of 0·1 *N* NaOH and a few ml of H_2O_2 solution are added, thus oxidising all the sulphur to SO_4''. After boiling to destroy excess of H_2O_2 the excess NaOH is back-titrated with 0·1 *N* H_2SO_4. From the equation—

$$Na_2S_x + (2x-2)NaOH + (3x-1)H_2O_2 \rightarrow xNa_2SO_4 + 4x\,H_2O$$
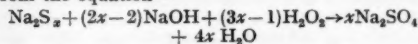
the amount of polysulphide is calculated.

To determine the sulphide sulphur, an aliquot part of the stock solution is slowly and with shaking run into an excess of acidified 0·1 *N* I soln. The excess of I is titrated with thiosulphate. One ml I soln. $\equiv$ 1·603 mg sulphide sulphur. The alkali content is determined by adding an excess of 0·1 *N* H_2SO_4, heating to boiling until the S coagulates, and titrating the excess NaOH. From—

$$Na_2S_x + H_2SO_4 \rightarrow H_2S + (x-1)S + Na_2SO_4$$
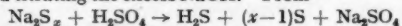
it is found that 1 ml 0·1 *N* $H_2SO_4 \equiv$ 2·2997 mg Na. Specimen analyses of Na disulphide and K pentasulphide are given. W. GOOD

72. On the volumetric determination of hydrogen sulphide and soluble sulphides. P. O. Bethge (*Anal. Chim. Acta*, 1953, **9** [2], 129–139).—Errors in determining sulphides by reaction with I are caused by the presence of ppt., air, or other ions. The method used allows the addition of I to the acidified sulphide *in vacuo*. Reagents alternative to I are

hypochlorite and $KMnO_4$. Results with hypochlorite are not so accurate as with iodine, but hypochlorite can be used in a micro method as it is more sensitive. Accuracy can be improved by standardisation of conditions and applying an empirical correction. $KMnO_4$ is not recommended. W. C. WAKE

73. The application of thiosalts in analysis. I. Complete precipitation of sulphides. I. K. Taimni and R. P. Agarwal (*Anal. Chim. Acta*, 1953, **9** [2], 116–120).—Whilst more commonly occurring ions of the copper and arsenic groups are easily pptd. by H_2S from acid solution, the pptn. of As (when present as AsO_4'''), Mo, and Pt is usually slow and incomplete. Se, although easily pptd., frequently forms an intractable vitreous mass. The alternative procedure investigated is the formation of thiosalts by conc. ammonium sulphide and their subsequent decomposition by HCl, thus immediately precipitating the metal sulphides. Other metals of the group also form thiosalts with the exception of Sn^{II}. Vanadium, although not normally pptd. by H_2S, becomes a member of the group when this procedure is adopted. The ppt. of Se is easily soluble in NH_4 sulphide. W. C. WAKE

74. The application of thiosalts in analysis. II. Estimations based on decomposition of thiosalts. I. K. Taimni and R. P. Agarwal (*Anal. Chim. Acta*, 1953, **9** [2], 121–128).—Quant. estimations of As, Se and Te based on weighing of the sulphides obtained as in the previous abstract. The compounds weighed are As_2S_5, SeS_2 and TeS_2; the last, it is claimed, gives the first reliable gravimetric method for Te. W. C. WAKE

75. Chemistry of selenium and its compounds. I. Iodometric method for determination of small quantities of selenium by cyanogen bromide. E. Schulek and E. Körös (*Z. anal. Chem.*, 1953, **139** [1], 20–24).—Elementary Se (4 to 40 mg) is heated to dryness with 0·2 g KCN, 1 drop of 96 per cent. alcohol and 10 to 15 drops of water on a bath of boiling water. The reagents are added again and the treatment is repeated. The KCNSe formed is dissolved in water and made up to 100 ml. Five ml of this solution, 70 ml of water and 1 g of boric acid are placed in a flask and boiled for 10 min.; the solution is cooled and transferred to a 200-ml Erlenmeyer flask. Eighty ml of water are added and the solution is neutralised with 0·1 *N* HCl and 1 drop of 0·2 per cent. ethoxychrysoidine solution as indicator. Saturated Br water is added until the solution is yellow, and after a few min. 3 to 5 ml of 5 per cent. phenol are added, followed by 2 to 3 ml of 20 per cent. HCl and 0·2 g of KI after a 5 min. interval. The liberated I is titrated with 0·1 *N* $Na_2S_2O_3$ and starch as indicator. 1 ml 0·1 *N* $Na_2S_2O_3 \equiv$ 0·3948 mg Se. With SeO_2 and SeO_3'', a quantity of the compound containing between 4 to 40 mg Se is dissolved in HCl and reduced with 5 per cent. hydrazine hydrate. The grey Se is converted to KCNSe by reaction with 0·2 g KCN, 2 ml of 96 per cent. alcohol and 20 to 40 ml of water, with continuous stirring. The treatment is repeated and the method then follows that for elementary Se. G. P. COOK

76. Qualitative detection of tellurium in tellurium-lead alloys. E. G. Brown (*Analyst*, 1953, **78**, 623–624).—A suitable test for Te in Te-Pb alloys is described. Into a hole in the cleaned surface of the metal a mixture of acetic acid and H_2O_2 (prep. described) is introduced. After reaction the turbid liquid is treated in a micro test tube with NaCl and

HCl, heated until clear and then treated with $SnCl_2$. A greyish-black coloration of colloidal Te is formed. Selenium acts similarly but does not occur in lead alloys.　　　　　　　　　　　A. O. JONES

77. Modified procedure for dissolving chromite ores. M. H. Khundkar and S. J. Quadir (*Analyst*, 1953, **78**, 623).—Many of the procedures for fusion of chromite ores are laborious and may lead to attack on the material of the crucible. Fusion with $KHSO_4$ or, better still, with a mixture of H_2SO_4 and K_2SO_4 yields highly satisfactory results, probably owing to formation of $K_2S_2O_7$ *in situ*. The fusion can be effected in a stout Hysil-glass beaker without inclusion of SiO_2 from the glass.　　　　　　　　　　　A. O. JONES

78. Colorimetric determination of molybdenum with di-sodium 1:2-dihydroxybenzene-3:5-disulpho-nate. F. Will and J. H. Yoe (*Anal. Chim. Acta*, 1953, **8** [6], 546–557).—This reagent (Tiron) gives a yellow colour with Mo with an absorption max. at $390 \, m\mu$. Beer's law is followed over the range 0·2 to 10 p.p.m. W causes interferences at $390 \, m\mu$, so when it is present Mo is determined from absorption at $420 \, m\mu$. Mo is pptd. by α-benzoinoxime, filtered, and dissolved in NH_3 solution, any remaining ppt. being washed through with acetone. This solution is used directly for the absorption measure-ments.　　　　　　　　　　　W. C. WAKE

79. Ultra-violet absorption spectrum of molyb-denum thiocyanate complex. G. E. Markle and D. F. Boltz (*Anal. Chem.*, 1953, **25** [8], 1261–1262).—An investigation into the ultra-violet absorption spectra of the molybdenum thiocyanate complex shows a characteristic ultra-violet absorbancy maximum suitable for determining small amounts of Mo. *iso*Butyl alcohol is recommended for ex-tracting the molybdenum thiocyanate complex for maximum development of absorbancy and hydra-zine sulphate as reducing agent, because acid stan-nous chloride interferes with ultra-violet absorbancy measurements. Interferences from ferric, ferrous, titanyl, and vanadate ions limit the usefulness of the method. Eleven solutions (4 p.p.m. of molyb-denum) gave a mean absorbancy value of 0·539 with a standard deviation of 0·0033. The ultra-violet spectrophotometric method with thiocyanate is more sensitive than the ultra-violet method with the peroxymolybdic acid complex.　　O. M. JONES

80. Colorimetric determination of uranium with dibenzoylmethane. J. H. Yoe, F. Will III, and R. A. Black (*Anal. Chem.*, 1953, **25** [8], 1200–1204).—A yellow complex is formed between U^{VI} and dibenzoylmethane, giving a maximum absorbancy at $395 \, m\mu$; Beer's law is obeyed over the concentra-tion range of 1 to 10 p.p.m. The 1 per cent. reagent is dissolved in 95 per cent. alcohol and the ethyl alcohol content of the final solution is 57 per cent. by vol. Optimum pH conditions are between 6·5 and 8·5 and the molecular ratio of reagent to $UO_2^{··}$ is 2 to 1. The sensitivity of the method is 0·05 p.p.m. in 1-cm cells. The most serious interference is from Fe, separation being accomplished by extraction of the U into ether. The limiting concn. for 32 metals and radicles is given. G. P. COOK

81. Separation of traces of the rare earth elements from uranium by means of amberlite IR-120 resin. D. Dolar and Zorica Draganić (*Rec. Trav. Inst. Rech. Structure Mat.*, 1953, **2**, 77–83).—Traces of rare earth elements are separated from 10-g samples of uranium nitrate by adsorption on a column

(height, 20 cm; diameter, 2 cm; rate of flow, 1 ml/min/sq. cm) of Amberlite IR-120 resin. The uranium is eluted with 1 to 1·5 litres of N oxalic acid and the rare earths with $\simeq$ 800 ml of 5 N HCl. ^{152}Eu nitrate was used as radioactive indicator in these studies; elution curves for U, Ce, and Eu are given.　　　　　　　　　　　W. MANZ

82. A study of the complex formation of salicyl-amide with uranyl ion and a colorimetric method for the estimation of uranium. A. K. Chakraburtty, D. Sen and P. Râÿ (*J. Indian Chem. Soc.*, 1953, **30** [7], 491–495).—A method for the estimation of U as the yellow UO_2 - salicylamide complex is des-cribed. Max. colour development occurs at pH 6·6 to 7·2 and absorbancy measurements are made in the 430 to 440 $m\mu$ region. The limit of detection is 2 μg. Beer's law is obeyed over a concentration range of 10 μg to 1·5 mg of U per ml. Interference of most common ions except $Fe^{··}$ and $Fe^{···}$ has been investigated. PO_4''', F', C_2O_4'', citrate, tartrate, Th'''', Ce'''', vanadate, molybdate, Pb'', Zn'' and Al''' interfere to a slight extent. Acetate interferes by forming complexes. The constitution of the uranyl complex is shown to be $UO_2[OC_6H_4.CONH_2]_2$ and its instability constant pK is determined as 11·37.　　　　　　　　　　　C. G. TAYLOR

83. The use of barbituric acid for the photometric determination of chlorine in water. E. Asmus and H. Garschagen (*Z. anal. Chem.*, 1953, **138** [6], 404–414).—The photometric determination of Cl in H_2O by use of barbituric acid in pyridine is nearly specific for Cl. Concn. of Cl from 1 μg/litre to 2·5 mg/litre can be determined in waters of pH between 2 and 10. One ml of 1 per cent. KCN solu-tion is added to 25 ml of the sample, and then after shaking, 3 ml of the barbituric acid reagent are im-mediately added. After 5 min., the intensity of the colour, whose absorption maximum lies at 578 $m\mu$, is determined by means of a Hg lamp. The optical measurement must be completed within 15 min. of the addition of the reagent. The reagent is pre-pared by dissolving 3 g of barbituric acid in 15 ml of pyridine, diluting with H_2O, adding 3 ml of conc. HCl and making up to 50 ml.　　　　J. H. WATON

84. Argentometric determination of bromide with nitroferroin as redox indicator. E. Rancke-Madsen (*Acta Chem. Scand.*, 1953, **7** [5], 741–744).—A new argentometric determination of bromide is described, with nitroferroin as a redox indicator. A strongly acidified aq. solution of nitroferroin, KBr and bromine water, all in low concn., is titrated with aq. $AgNO_3$ until the red colour has virtually disappeared. The sample in which bromide is to be determined is added, and titrated with aq. $AgNO_3$ until the ppt. changes in colour from red to yellow. The mean of ten determinations on a KBr solution prepared 0·09988 M was 0·09997 M showing that the method yields results 0·1 per cent. too high. An attempt at following the titration potentiometrically was un-successful. Chloride or iodide must be absent.　　　　　　　　　　　A. R. MATHIESON

85. Determination of bromine in liquids by X-ray fluorescent spectroscopy. G. T. Kokotailo and G. F. Damon (*Anal. Chem.*, 1953, **25** [8], 1185–1187).—The errors caused by impurities and variations in base stock are overcome by application of Se as an internal standard in the determination of Br in hydrocarbons. The standard consists of 0·54 per cent. by weight Se as ethyl selenide in a $(1 + 1)$ v/v mixture of benzene and decane, 10 ml being added to 50 ml of sample. The ratio of bromine to internal

standard fluorescence is determined as a function of the Br content and this ratio is unaffected by impurities. For samples containing 0 to 0·4 per cent. by wt. of Br the error is $\pm 0\cdot01$ per cent.

G. P. Cook

86. Determination of iodine and bromine in mineral waters by means of differential titration. G. P. Alexandrov and T. F. Levtchenko (*Ukr. J. Chem.*, 1951, **16**, 599–611).—A summary of existing methods for determination of iodine and bromine in naturally occurring compounds and mineral waters is given. Bromine was determined in standard solutions of salts of bromine, then in natural mineral waters and by addition of bromine to the latter. The influence of sodium chloride on the accuracy of determination of bromine was found to be negligible. On treatment of samples with hypochlorite, the iron contained in mineral waters separated as hydroxide. The influence of the acidity of the medium on the reactions of IO_3' and I', BrO_3' and I': the acidity of the solution in which IO_3' and I' would react and BrO_3' and I' not, corresponds to a concentration of hydrogen ions in which a solution of methyl orange keeps its red coloration at pH 3·1.

Differential titration: A saturated solution of NaCl (10 ml) is added to a mixture of standard solutions of mineral waters containing I and Br (50 ml), followed by potassium hypochlorite (10 ml), and the mixture is heated to 80° C. Boric acid (10 ml) is added and heating is continued for 5 min.; to this solution 4 per cent. H_2O_2 (20 ml) is added and heated for a further 15 to 20 min. $0\cdot5 N$ H_2SO_4 (0·5 ml) is added to the solution followed by a 0·2 per cent. solution of starch (1 ml) and 10 per cent. solution of KI (4 ml); the iodine is titrated with 0·005 N solution of thiosulphate. Bromine is determined after iodine: to the solution 3 N H_2SO_4 (15 ml) is added, 10 per cent. KI (6 ml), ammonium molybdate, and titration is carried out with a 0·005 N solution of thiosulphate.

E. Premuzic

87. Micro-determination of iodides by arresting the catalytic reduction of ceric ions. B. Rogina and M. Dubravčić (*Analyst*, 1953, **78**, 594–599).—The basis of the method described for determining 0·01 to 1 μg of I as I' is the catalytic effect of iodides on the reduction of Ce^{IV} salts by As_2O_3. To avoid technical difficulties in measuring the concn. of unreduced Ce^{IV} ions while the reaction is in progress, the reaction is arrested by addition of excess of Fe^{II} salt. $Fe^{\cdots}$ ions are formed equivalent to the unreduced $Ce^{\cdots\cdot}$ ions and the concn. of these after addition of KCNS is determined photometrically; the corresponding concn. of iodide is deduced from a calibration graph.

A. O. Jones

88. Volumetric determination of manganese and chromium using silver peroxide. M. Tanaka (*Bull. Chem. Soc. Japan*, 1953, **26** [6], 299–302).—The reagent is stable, oxidation of both elements is rapid. The reagent is suspended in water and added to a solution containing Mn or Cr N in H_2SO_4. Excess of peroxide is destroyed by heating on a water-bath for 2 min.; the solution is then titrated with 0·02 N oxalate (for Mn) or with $KMnO_4$ after addition of excess of $FeSO_4$ (for Cr). The results obtained for Cr compare favourably with those by permanganate, and for Mn with those obtained by the persulphate method.

C. D. Lafferty

89. Titration of a ferric salt with ethylenediaminetetra-acetate. D. Lyndersen and O. Gjems

(*Z. anal. Chem.*, 1953, **138** [4], 249–256).—The dependence of the results of the titration of Fe^{III} with di-sodium ethylenediaminetetra-acetate with thiocyanate as indicator is theoretically and experimentally investigated. The plot of the vol. of reagent required against log (concn. of indicator) shows a minimum, the vol. co-ordinate of which is too small by an amount equivalent to the Fe^{III}-indicator complex. As the concn. of the complex can be made very small the minimum can be used to determine a suitable indicator concn. To improve the visibility of the end-point the system is shaken with an organic solvent (ether) to give a layer in which the indicator colour is more easily observed. The undesirable effect produced by the presence of $Al^{\cdots}$ and $Zn^{\cdots}$ of slowing down the rate at which the reaction equilibrium is established is eliminated by addition of citrate.

W. Good

See also Abstracts 2, 3, 4, 5, 127, 128, 134, 171, 173, 195, 199.

3.—ORGANIC ANALYSIS

90. Determination of carbon and hydrogen in organic substances containing sulphur and nitrogen in presence of catalysts. III. P. N. Fedoseev and M. M. Pavlenko (*J. Anal. Chem., U.S.S.R.*, 1953, **8**, 158–162).—The authors' method (*Brit. Abstr. C*, 1951, 33 and 1952, 83) for determining C and H is extended to cover the analysis of organic substances containing S and halogens in addition to C, H, O, and N. Removal of S oxides and halogens is attained by the use of metallic Ag at 650° to 750°C.

G. S. Smith

91. Determination of vapours of fluoro-organic compounds in air. E. A. Peregud and B. S. Boykina (*J. Anal. Chem., U.S.S.R.*, 1953, **8**, 178–181).—Air containing a F-organic compound is passed through a quartz tube at 900° C, the SiF_4 formed is absorbed in water and the Si content is determined by means of the Mo-blue reaction by means of ascorbic acid.

G. S. Smith

92. Ternary methanol - ethanol - acetone and three related binary solutions. Refractive indices and bubble points. H. H. Amer, R. R. Paxton, and M. Van Winkle (*Anal. Chem.*, 1953, **25** [8], 1204–1206).—A method is given which makes it possible to find the composition of any methanol - ethanol - acetone solution for which the refractive index and bubble point are known.

G. P. Cook

93. Some applications of the Komarowsky reaction. S. D. Nogare and John Mitchell, jun. (*Anal. Chem.*, 1953, **25** [9], 1376–1379).—A colorimetric procedure for the determination of 1 : 2-propylene glycol in the presence of ethylene glycol is described. An aq. solution of the sample containing up to 0·2 mg of 1 : 2-propylene glycol and < 10 mg of ethylene glycol is treated with 30 $\pm$ 1 mg of p-hydroxybenzaldehyde; 5 ml of conc. H_2SO_4 is added and the mixture is heated in boiling water for 30 min. The solution is then diluted to a suitable volume with dil. $(1 + 1)$ H_2SO_4, the absorbancy being measured at 540 mμ against a reagent blank after 1 hour. The 1 : 2-propylene glycol content is determined from a calibration curve. Other compounds giving a positive reaction with p-hydroxybenzaldehyde are acetone, ethyl methyl ketone, ethyl *iso*propyl ketone, *cyclo*hexanone, acetaldehyde and propionaldehyde. Application of the method to the determination of *iso*valeraldehyde in succinic

acid gives accurate results over the concn. range of 0·01 to 0·1 mole per cent. of aldehyde in acid.

G. P. COOK

94. Periodic acid, a functional reagent for organic analysis. M. P. Fleury (*Chim. Anal.*, 1953, **35** [9], 197–206).—The oxidation of oxygenated organic compounds by periodic acid is reviewed in detail. Each secondary alcohol group of a polyhydric alcohol gives a molecule of formic acid whilst primary alcohol groups become CH_2O. The terminal aldehydic group of aldoses gives formic acid, methylketones give acetic acid. The general condition for a substance to be attacked by periodic acid in dilute solution is that it should contain at least two contiguous C atoms each having OH attached (methylketones are apparently included as enols). Exceptions exist when an active methylene group separates the hydroxylated carbons. Thus such a compound as $CH_2OH.CH_2.CH_2OH$ is not attacked, but $COOH.CH_2.COOH$, the CH_2 group of which is reactive, is attacked, giving two molecules of formic acid and CO_2.

The analytical problems considered are those of glycerol and the glycols, the polyalcohol acids, and the α-amino acids. The determination of the consumption of periodic acid can be made either by reaction with KI in a buffered solution of pH 7 to 8 (in which HIO_3, the product of the reduction of periodic acid, does not react with KI but HIO_4 does) or by reaction with KI in acid solution, allowance being made for the iodine liberated by HIO_3. The former method is preferable especially if the determination is made by the addition of an excess of As_2O_3. Alternative analytical procedures are outlined for the determination of the products, and the problem of glycol mixtures is discussed with critical details. Tartaric and gluconic acids are used as examples of polyalcohol acids. A selected bibliography of 6 entries is given.   W. C. WAKE

95. Determination of formic acid in presence of formaldehyde bisulphite. L. Ahlén and O. Samuelson (*Anal. Chem.*, 1953, **25** [8], 1263–1264).—A method described for determining formic acid in the presence of formaldehyde bisulphite is used in the analysis of sulphite waste liquor. The SO_2 is removed by precipitation as barium sulphite and the formic acid and formaldehyde are subsequently separated by means of an acetate ion-exchanger. After elution, the acid is determined according to the mercuric chloride method. Tabulated experimental results show that added and found amounts of formic acid agree well and that the maximum relative error is 1·4 per cent. Similar accuracy has been attained with solutions containing excess of acetic acid as well as formic acid.   O. M. JONES

96. Separation of maleic and malonic acids by paper chromatography. J. W. Airan and J. Barnabas (*Naturwissenschaften*, 1953, **40**, 510).—Mixtures of tartaric, citric, maleic, malonic, malic and succinic acids are separated on (*i*) paper strips, (*ii*) one dimensional circular paper, and (*iii*) two dimensional circular paper chromatograms (Airan, *Brit. Abstr. C*, 1953, 280) from a pentanol–formic acid solvent. Separation of maleic and malonic acids only occurs in (*ii*) and (*iii*), the latter giving a more distinct separation.   D. R. GLASSON

97. Stability in air of solutions of oxalates. E. Abel (*Z. anal. Chem.*, 1953, **138** [4], 257–258).—It is shown why oxalate solutions must be kept free from traces of organic matter to ensure stability.

W. GOOD

98. The dielectric constant and salt effects upon the acid hydrolysis of methyl propionate. J. L. Hockersmith and E. S. Amis (*Anal. Chim. Acta*, 1953, **9**, [2], 101–110).—Variation in dielectric constant is produced by progressive addition of acetone to the aq. solution and ionic concn. is varied by addition of $NaNO_3$. In regions of high dielectric constant the plots of log k/C_{H_2O} against $1/E$ are straight lines as expected from the theory of reactions between positive ions and dipolar molecules. Instead of calculating the slope from known molar radii the reverse is done, giving values supporting the theory. In the investigation of the salt effect, the activities of the hydronium ions were assumed by calculation from the Debye-Hückel approx. equation and hence the ionic strength was assessed. The reaction velocities found indicate a water catalysed minimum rate, showing that the reaction is catalysed generally by acids or bases. The salt effect depends on the effect of the ionic strength on the hydronium ion from a completely dissociated strong acid (HCl).   W. C. WAKE

99. The dielectric constant effect upon the acid hydrolysis of ethyl acetate. P. M. Nair and E. S. Amis (*Anal. Chim. Acta*, 1953, **9** [2], 111–115).—A specific solvent effect exists in the hydrolysis of ethyl acetate by HCl; this masks the positive ion interaction with the dipole. This contrasts with the methyl propionate hydrolysis reported in the previous abstract.   W. C. WAKE

100. Determination of diacetyl. D. T. Englis, E. J. Fisch and S. L. Bash (*Anal. Chem.*, 1953, **25** [9], 1373–1375).—A sample containing 0·1 to 1·0 mg of diacetyl is treated with 20 ml of 10 per cent. $NH_2OH.HCl$ and 10 ml of 40 per cent. Na acetate and heated for 1 hour at 85° C or set aside overnight at room temp. The absorbancy of this solution, after suitable dilution, is measured at 226 mμ against a reagent blank as reference solution. The diacetyl content is found by reference to a suitable calibration curve prepared from pure dimethylglyoxime; concentrations between 0 and 10 p.p.m. are easily determined. Results by this method compare well with those by the gravimetric procedure in which Ni dimethylglyoxime is formed; the method is applicable to the direct determination of diacetyl in distillates from food products.   G. P. COOK

101. Paper chromatography of aliphatic amines. J. Bertetti (*Ann. Chim., Roma*, 1953, **43**, 361–364).—The R_F values of 37 primary, *sec.*, and *tert.* aliphatic amines and polyamines are determined in 25 per cent. acetic acid - *n*-butyl alcohol as the mobile phase. The R_F values, which are tabulated, vary with the number of C atoms in amines of homologous series. It is confirmed that primary monoamines, like *sec.* amines and many polyamines, give colorations with ninhydrin in the same way as amino acids. The *tert.* amines give only slight colorations or none at all.   C. A. FINCH

102. Systematic method for identification of amines, using colour reactions. I. Aliphatic amines. J. Bertetti (*Ann. Chim., Roma*, 1953, **43**, 351–360).—A systematic method for the identification of aliphatic amines, based on four colour reactions, is described. The validity of the method is shown for 42 amines; it is possible to distinguish between isomers. A small amount of amine in 1 per cent. solution in C_2H_5OH is sufficient for the reactions.

The following reagents are used: (*i*) aqueous Na nitroprusside (1 per cent. w/v), with C_2H_5CHO (2 ml) and 95 per cent. C_2H_5OH (5 ml), kept for 48 hr.

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before use (the solution keeps for $\simeq$ 2 months); (ii) as (i), but with CH_3CHO instead of C_2H_5CHO; (iii) saturated aq. $HgCl_2$; (iv) quinone (0·5 per cent.) in C_2H_5OH (95 per cent.). The amine to be examined is treated with each of these reagents. The colours obtained are tabulated. C. A. FINCH

103. **Determination of nitrogen in nitrocellulose and of nitrocellulose and combined phthalate in lacquers.** W. E. Shaefer and W. W. Becker (*Anal. Chem.*, 1953, **25** [8], 1226–1231).—A quantitative ferrous chloride reduction method described for measuring the nitrate nitrogen in nitrocellulose is directly applicable to the determination of nitrocellulose in a lacquer assuming that the lacquergrade nitrocellulose contains 12·0 per cent. of nitrate nitrogen. In the method described for determining the combined phthalate content of an alkyd resin in a nitrocellulose lacquer, the nitrocellulose is converted into a water-soluble form by treating the lacquer solution in acetic acid with ferrous chloride, the alkyd resin is extracted with methylene chloride, and the phthalate content is determined by a modified Kappelmeier procedure. A further modification of the Kappelmeier method for determining phthalate in the presence of diglycollate is described. Some results are presented. O. M. JONES

104. **Determination of acrylonitrile in coloured solutions.** G. J. Janz and N. E. Duncan (*Anal. Chem.*, 1953, **25** [9], 1410–1411).—A modification of the standard method for acrylonitrile is described. After reaction of the nitrile with dodecyl mercaptan in the presence of a basic catalyst, the excess of mercaptan is determined potentiometrically by means of a silver electrode and with 0·1 N $AgNO_3$ in a mixture of *iso*propyl and ethyl alcohols as the titrant. This method is capable of the same precision and accuracy as the standard iodimetric method. G. P. COOK

105. **Comparison of qualitative tests for mercapto-compounds.** Hans Freytag (*Z. anal. Chem.*, 1953, **138** [4], 259–266).—The sensitivities of known tests for thioglycollic acid, mercaptobutanesulphonic acid and cysteine and of those with alkali molybdate, lead acetate and cupric ammonium chloride as test reagents are compared. The most sensitive is Fleming's modification of the sodium nitroprusside test. W. GOOD

106. **Two techniques for determination of organic sulphides.** W. H. Houff and R. D. Schuetz (*Anal. Chem.*, 1953, **25** [8], 1258–1259).—Two methods for determining organic sulphides are described. In the back-titration method, excess of a bromine - bromate solution is added and the excess determined iodimetrically. The method cannot be applied to dialkyl sulphides which are easily oxidised to sulphones by excess of bromine. Aromatic and heterocyclic sulphides including slightly coloured ones are determined by treating them in acetic acid solution with a slight excess of bromide - bromate solution and 3 ml conc. HCl and back-titrating the iodine with standard thiosulphate solution. In the dead-stop end-point method used for determining any organic sulphide by bromine oxidation, the end-point is indicated by a current flowing between two Pt electrodes. Both the precision and accuracy are high (within 0·5 per cent.). Results by both methods are tabulated. O. M. JONES

107. **The use of paper chromatography for the separation and characterisation of mesoinositol and scyllitol.** P. Fleury, J.-E. Courtois and P. Malangeau (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [5–6], 537–540).—The two most frequently encountered isomeric inositols (*meso*inositol and scyllitol) have been separated by paper chromatography, by means of a developer such as 90 per cent. v/v methanol and, after drying, by a more dilute alcohol solution or by Partridge's mixture. The separated spots are revealed by the colour produced by $Hg(NO_3)_2$ in HNO_3. The other isomers appear to be equally displaced and they adopt an intermediate position between *meso*inositol and scyllitol. J. G. HEATHCOTE

108. **Separation of phenols by partition chromatography.** T. R. Sweeney and J. D. Bultman (*Anal. Chem.*, 1953, **25** [9], 1358–1363).—Mixtures of phenols are separated by partition chromatography with water as the immobile phase and *iso*octane as the mobile phase, silicic acid being used as the support. Resolution of the mixture is achieved by spectrophotometric examination. The proportion of water to silicic acid is critical with respect to the rate of movement of different phenols through the column and to the diffusiveness of their respective zones in the column; therefore by running several chromatograms with various proportions of water the resolution of a given mixture is enhanced. G. P. COOK

109. **Determination of volatile phenols in cigarette smoke.** C. H. Rayburn, W. R. Harlan and H. R. Hanmer (*Anal. Chem.*, 1953, **25** [9], 1419).—Cigarette smoke is collected according to the technique of Bradford, Harlan and Hanmer (*Ind. Eng. Chem.*, 1936, **28**, 836) and the solution is steam distilled. The distillate is extracted with ether, which is then extracted with NaOH; the alkaline solution is then neutralised and finally steam distilled. Aliquots of the distillate at 0° C are treated with p-nitroaniline and $NaHCO_3$, then neutralised, extracted with ether and the residue is dissolved in acetone after evaporation of the ether. Chromatographic separation on Na_2CO_3-impregnated paper is carried out with ether - light petroleum as the solvent, followed by elution of the various bands with *iso*propyl alcohol and measurement of absorbancies. Measurement for guaiacol and the cresols is at 510 mμ and for phenol at 490 mμ. The phenol content is calculated from the values obtained from standards. Maximum variation between duplicates is $\pm$ 5 per cent. for guaiacol and cresols and $\pm$ 2 per cent. for phenol. G. P. COOK

110. **Ultraviolet analysis of isomeric cresol mixtures.** G. E. Carney and J. K. Sanford (*Anal. Chem.*, 1953, **25** [9], 1417–1418).—Mixtures of o-, m- and p-cresols in *iso*octane are measured accurately at 2728, 2774, 2858 and 3000 A against *iso*octane at spectral band widths of 5A. The concn. of each isomer is calculated from simultaneous equations involving the total absorbancy at each λ and the ratios of the absorbances of the isomers at the different wavelengths. Phenol and alkylphenols interfere. The largest error obtained is 1·6 per cent. (absolute) and standard deviations for o-, m- and p-cresol were 0·736, 1·03 and 0·283 per cent. respectively. G. P. COOK

111. **Spectrophometric determination of phthalic acid in phthalic anhydride.** M. M. Agarwal and F. Spagnolo (*Anal. Chem.*, 1953, **25** [9], 1412–1413).—The phthalic acid content of the sample is separated by dissolving the phthalic anhydride in $CHCl_3$,

leaving the residue of the acid. This is dissolved in 0·1 N NaOH, the solution is neutralised to pH 2·0 with 3 N HCl and the absorbancy of appropriate dilutions with 0·1 N HCl is measured at 276 mμ against a blank of 0·1 N HCl. The phthalic acid content is determined by reference to a suitable calibration curve. If the determination of phthalic anhydride is also required, the absorbancy of the $CHCl_3$ solution is measured at 291 or 300 mμ against a blank of $CHCl_3$. The standard deviation of a single determination for the acid is 0·045 per cent. and precision for the anhydride method is ± 0·5 per cent.

G. P. Cook

112. Spectrophotometric determination of anthraquinone and benzanthrone. K. B. Whetsel (*Anal. Chem.*, 1953, **25** [9], 1334–1337).—The sample is dissolved in conc. H_2SO_4 and suitably diluted; the absorption of this solution is measured at 266 and 515 mμ against conc. H_2SO_4 as reference liquid. The cell correction is determined by measuring the absorbance at the analytical wavelengths with pure solvent in each cell. The percentages of anthraquinone and benzanthrone are calculated from these measurements, the benzanthrone alone being responsible for the absorbance at 515 mμ; the absorbance of the anthraquinone is corrected at 266 mμ for benzanthrone absorbance at this λ by previous determination of the ratio of its absorbances at the two λ used. Beer's law is followed and the largest deviation for the benzanthrone determination is 1·67 per cent. and for the anthraquinone 0·64 per cent. Accuracy is 100 ± 1·5 per cent.

G. P. Cook

113. Determination of small amounts of *m*-dinitrobenzene in nitrobenzene. F. G. Angell (*Analyst*, 1953, **78**, 603–606).—Small amounts of *m*-dinitrobenzene in nitrobenzene can be adsorbed chromatographically on Al_2O_3. The nitrobenzene is eluted with a mixture of benzene and light petroleum. The column is then extruded in separate sections, which are dried, and the adsorbed *m*-dinitrobenzene is extracted with ethanol. With conc. > 0·02 per cent. the *m*-dinitrobenzene is determined polarographically and with smaller amounts absorptiometrically after reaction with acetone and NaOH.

A. O. Jones

114. Infra red spectra for analysis of aldehyde and ketone 2:4-dinitrophenylhydrazones. J. H. Ross (*Anal. Chem.*, 1953, **25** [9], 1288–1303).—Identification of aldehydes and ketones is achieved by infrared examination of their 2 : 4-dinitrophenylhydrazones; closely related aldehydes and ketones differing by one CH_2-group exhibit markedly different spectra. The derivative is examined as a paste on a NaCl window, a range of 6 to 15 μ being covered with a NaCl prism. Identification is possible with as little as 10 mg of derivative and the method is applicable to quant. determination of a mixture of small amounts of acetaldehyde and propionaldehyde in water. Diagrams of 37 spectra for numerous aldehyde and ketone derivatives are given.

G. P. Cook

115. Photometric determination of pyridine in water and ammoniacal solutions. E. Asmus and H. Garschagen (*Z. anal. Chem.*, 1953, **139** [2], 81–89).—In the procedure for water, 2 ml of 0·1 N HCl, 1 ml of 1 per cent. KCN and 5 ml of 1 per cent. chloramine solution are shaken together. Five ml of the sample are added, the solution is mixed, and set aside for exactly 5 min. Ten ml of 1 per cent. barbituric acid are added with continuous shaking and the absorbancy of the reddish-violet colour is

measured after 30 min. at 578 mμ against water. The pyridine content is found by reference to a suitable calibration curve. For ammoniacal solutions of pyridine, the sample containing from· 4 to 3400 μg of pyridine is placed in a 100-ml measuring cylinder and neutralised approximately with 25 per cent. HCl. Two drops of 0·1 per cent. *p*-nitrophenol are added and the solution is titrated with 0·1 N HCl until colourless. After cooling, the solution is made up to 100 ml with water, and 5 ml of this solution is treated as for the determination of pyridine in water. As little as 0·25 μg of pyridine can be determined by this method.

G. P Cook

116. Paper chromatography of pyrazolone derivatives. W. Dihlmann (*Naturwissenschaften*, 1953, **40**, 510–511).—Pyrazolone derivatives are characterised by determining their R_F values by use of matted filter-paper and a solvent mixture of butyl alcohol, acetic acid and water. The zones are identified by colorations with Ehrlich's reagent, which is $(p\text{-}(CH_3)_2N.C_6H_4.CHO$ in HCl).

D. R. Glasson

117. Preparation and properties of some methylated indans. J. Entel, C. H. Ruof and H. C. Howard (*Anal. Chem.*, 1953, **25** [9], 1303–1310).—A number of indans were synthesised by conventional methods and purified by fractional distillation in columns of 50 theoretical plates. Physical measurements made on the indans were of infra-red and u.v. spectra, boiling points, densities and refractive indices. The indans prepared were the 1-methyl, 2-methyl, 4-methyl, 5-methyl, 1 : 2-dimethyl, 1 : 3-dimethyl, and 1 : 6-dimethylindans. The infra-red spectra appear to give an effective means of identifying methylated indans in mixtures of polymethylbenzenes having similar boiling points. G. P. Cook

118. Determination of mole ratio of urea to formaldehyde in their condensation products. P. P. Grad and R. J. Dunn (*Anal. Chem.*, 1953, **25** [8], 1211–1214).—For the determination of formaldehyde, the resin is hydrolysed with H_3PO_4 and the liberated formaldehyde distilled over into a measured volume of KCN solution. The excess of KCN is determined gravimetrically as AgCN, the percentage of formaldehyde being obtained by calculation from this result and from that of a blank test on the KCN. Halogen compounds are the most frequently occurring interfering substances, but hydrazine compounds also interfere. The urea is determined after reaction with benzylamine by separation and weighing of the dibenzylurea formed. Interfering substances are those containing, in addition to an amino group, a carbonyl or thiocarbonyl group. The max. deviation of the formaldehyde analysis is 0·08 per cent. with a recovery min. of 99·8 per cent. The urea determination gives a precision of 0·3 per cent. and a recovery of 100 ± 0·1 per cent; the recovery in fully cured resins varies between 90 and 99 per cent.

G. P. Cook

119. Determination of added hexamethylenetetramine in two-stage phenol-formaldehyde resins. E. H. Chipperfield and H. Busfield (*Analyst*, 1953, **78**, 617–619).—Hexamine, used to provide formaldehyde in the preparation of two-stage phenol-formaldehyde resins, is usually determined by titration of an extract of the finely divided resin with a strong acid. This involves a time-consuming filtration. In the conductimetric method described filtration is avoided. Presence of "free phenol" in amounts usually occurring in such resins (up to 6 per cent.) does not affect the titre.

A. O. Jones

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120. **Analysis of lacquer thinners by fluorescent indicator adsorption method.** W. H. Ellis and R. L. Le Tournea (*Anal. Chem.*, 1953, **25** [8], 1269–1270).—A rapid accurate adsorption method for the simultaneous direct determination of aromatics, saturates, total hydrocarbons, and total oxygenated compounds in lacquer thinners is described. Tabulated data indicate its accuracy. O. M. JONES

121. **Rapid determination of tetraethyl lead [in motor spirit] by means of complexone.** A. Grünwald (*Erdöl u. Kohle*, 1953, **6**, 550–551).—The 20 to 25-ml sample is pipetted into a 500-ml flask and Br soln. (30 per cent. w/w in CCl₄) is added until the colour persists. Methanol or ethanol is added until the ppt. (which contains org. matter that would vitiate a direct titration) and the two liquid phases form a clear homogeneous solution, which is boiled and decolorised with a small excess of N alcoholic KOH. Water (100 to 200 ml) is added and the two-phase liquid is refluxed gently. A measured vol. (x ml) of aq. di-sodium ethylenediaminetetra-acetate (6·635 mg per ml) previously standardised with CaCO₃ is added. The solution is acidified with aq. H₂SO₄ and neutralised to methyl red. Buffer solution (10 ml containing 54 g NH₄Cl and 80 g NH₃ per litre) and Eriochrome Black T (0·4 g per 100 ml of alcohol) are added and the solution is back-titrated with y ml of aq. ZnSO₄ (5·128 mg of ZnSO₄.7H₂O per ml) exactly equivalent in strength to the complexone solution. The tetraethyl lead content in ml per litre of sample is then $3·489(x - y)/\text{vol. of sample.}$ A. R. PEARSON

122. **Observations on the determination of iodine value with pyridine sulfate dibromide.** H. J. Lips (*J. Amer. Oil Chem. Soc.*, 1953, **30** [10], 399–403).—Values obtained by the Benham-Klee method (*cf. Brit. Abstr. C*, 1950, 449) for linseed and rape oils and their polymerisation products increase with the reaction time and the amount of reagent used, but, with attention to details, reproducible values can be obtained with a 300 to 400 per cent. excess reacting for 4 hr. The presence of the Hg acetate catalyst affects the keeping property of the blank, reduces the iodine values of the oils (probably through promoting oxidation at the double bonds), and gives high and erratic iodine values in the presence of S. In presence of CHCl₃, the use of the catalyst gives I val. that decrease with the reaction time. Values obtained by the Hunter-Hyde modification of the Wijs method (*cf.* A.O.C.S. Official Methods, 1946) tend to increase with prolonged reaction time, but are only slightly affected by the presence of S or CHCl₃. P. S. ARUP

123. **Halogenation method for the determination of the unsaturation of tung oils and of elaeostearic acid.** R. W. Planck, F. C. Pack and L. A. Goldblatt (*J. Amer. Oil Chem Soc.*, 1953, **30** [10], 417–419).—The following modifications of the Benham - Klee method (*Brit. Abstr. C*, 1950, 449) give for tung oil and for partly hydrogenated tung oil iodine values agreeing with those obtained by quantitative catalytic hydrogenation (*cf.* Pack *et al.*, *Brit. Abstr. C*, 1952, 443), and give theoretical values for α and β-elaeostearic acids; the Hg acetate solution is added to the solution of the sample (75 to 85 mg) in CCl₄ (5 ml) before (instead of after) the pyridine sulphate dibromide reagent (50 ml); reaction flasks of red glass are used, the reagents are measured out and added in semi-darkness, and the charged flasks are kept in complete darkness during the reaction period (1 hr). Normal lighting is used from the commencement of the actual titration. P. S. ARUP

124. **Modified procedure for the quantitative bromine absorption of oils and fats.** E. H. W. J. Burden and D. N. Grindley (*Analyst*, 1953, **78**, 619–621).—Certain disadvantages of the most widely used modification of the Becker method (Toms, *Brit. Abstr. B*, 1928, 236) for determining the Br absorption of oils and fats rendered it unsuitable, in spite of its speed and simplicity, for routine work in an investigation of the properties and estimation of stearolic acid. The diffusion method now described is applicable to a larger amount of oil (0·3 g), which is exposed to Br vapour for 30 min.; excess Br is removed by stirring at room temp. In presence of stearolic acid the time of exposure must be increased to 24 hr. and the excess Br removed at 110° C. A. O. JONES

125. **Identification of complex organic materials by infra red spectra of their pyrolysis products.** D. L. Harms (*Anal. Chem.*, 1953, **25** [8], 1140–1155).—The material is placed in a borosilicate test tube and dry distilled, the temp. of pyrolysis depending on the nature of the sample. The vaporous pyrolysate is condensed as a liquid on the cooler portion of the tube and is then transferred to a NaCl window. A "sandwich" is made by pressing a second window over the pyrolysate on the first window, and infra-red spectra are obtained in the usual manner. The method is applicable to many materials of rubber, plastic and resinous composition that are not adapted to conventional infra-red analysis because of their physical intractability. Spectra of 35 materials of this class are illustrated and the appropriate data for their reproduction is given. G. P. COOK

126. **Identification of polymeric materials.** P. F. Kruse, jun. and W. B. Wallace (*Anal. Chem.*, 1953, **25** [8], 1156).—The shredded or cubed polymeric material is dry distilled and the products of decomposition are collected in CCl₄. The infra-red spectrum of the pyrolysis products affords the means of identification. G. P. COOK

See also Abstracts 135, 139, 159, 196.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

127. **Determination of calcium in blood serum and biological fluids.** J.-A. Gautier and P. Pignard (*Ann. Pharm. Franç.*, 1953, **11**, 438–445).—The use of complexones for the determination of Ca·· is reviewed. In blood serum and cerebrospinal fluid, the Ca is liberated by bringing 2 ml of serum to pH ≯ 6 with 1 ml of 1 per cent. acetic acid, and then pptd. as oxalate. The ppt. is dissolved in 0·25 ml of N HCl. The tube is heated for a short time on a water-bath, and then cooled; 2 ml of 0·004 M complexone-III (di-sodium dihydrogen ethylenediaminetetra-acetate) are added. One ml of 15 N aq. NH₃ is added and the solution is back titrated with 0·002 M NiSO₄ until the murexide indicator changes colour from violet to a champagne-yellow (the end-point is much sharper than by back titration with Ca·· at this concn.). For urinary Ca, the method is similar, but the complexone solution is 0·04 M and back titration is with 0·01 M Ca·· solution. E. J. H. BIRCH

128. Improved spectrophotometric procedure for determination of serum iron using 4:7-diphenyl-1:10-phenanthroline. Ralph E. Peterson (*Anal. Chem.*, 1953, **25** [9], 1337–1339).—One to two ml of serum or plasma are diluted to 6 ml, treated with 2 ml of a 1 per cent. solution of thioglycollic acid in 20 per cent. trichloroacetic acid, heated in a water-bath at 90° to 95°C for 10 to 15 min., and centrifuged and the supernatant solution removed. The treatment is repeated on the ppt. using 0·5 ml of the thioglycollic acid reagent and 2 ml of water. The supernatant solutions are combined, 2 ml of saturated Na acetate and 2 ml of 0·0025 M 4:7-diphenyl-1:10-phenanthroline in *iso*amyl alcohol are added followed by extraction with 6 ml of *iso*amyl alcohol. An aliquot of the alcohol layer is taken, 0·5 ml of ethanol is added and the absorbancy is measured at 535 mμ. Reagent blanks and standards containing 2 to 4 μg of Fe are also tested. Precision of the method is $\pm$ 5 per cent. with 95 to 100 per cent. recoveries.   G. P. COOK

129. Determination of various amino-acids in blood plasma. T. J. H. Huisman (*Chem. Weekbl.*, 1953, **49**, 705–710).—The 60-ml blood sample containing added sodium citrate is centrifuged, and a protein-free plasma is prepared by ultrafiltration at 0°C through a cellophane membrane (Perkin Elmer Corpn.) at a pressure of 200 mm of Hg greater than atmospheric in an atmosphere consisting of 95 per cent. O and 5 per cent. CO_2; further directions to be observed are given. A previously described chromatographic method (*Brit. Abstr. C*, 1953, 314) is used. Results of analyses of a blood plasma and plasma hydrolysate from a normal adult are tabulated and compared with other published results.   P. S. ARUP

130. Method for determining 17-hydroxycorticosteroids in blood. D. H. Nelson and L. T. Samuels (*J. Clin. Endocrin.*, 1952, **12**, 519–526).—Laked blood (30 ml) is extracted with ether - $CHCl_3$ mixture. The dried extract is dissolved in ethanol, washed with hexane, dried, and redissolved in $CHCl_3$. Plasma (10 ml) is extracted with $CHCl_3$, dried, and redissolved. The $CHCl_3$ solution is passed through a Mg silicate - Celite or Florisil column. Elution is with ethanol - $CHCl_3$ mixtures. The eluates are incubated at 60°C with phenylhydrazine - H_2SO_4 and the 17-hydroxycorticosteroids are determined spectrophotometrically. One μg can be measured to $\pm$ 1 per cent., and 2 to 4 μg of 17-hydroxycorticosterone added to 30 ml of blood can be fully recovered.   P. C. WILLIAMS

131. Oxidation of corticosteroids with sodium bismuthate. C. J. W. Brooks and J. K. Norymberski (*Biochem. J.*, 1953, **55**, 371–378).—An analytical method for the estimation of corticosteroids is developed. The method is based on the estimation of formaldehyde and 17-ketosteroids following the oxidation of corticosteroids with sodium bismuthate. The released formaldehyde is estimated by its colour reaction with chromotropic and sulphuric acids (Eegriwe, *Z. anal. Chem.*, 1937, **110**, 22; *cf.* MacFadyen, *J. Biol. Chem.*, 1945, **158**, 107), and the 17-ketosteroids are assayed by the Zimmermann reaction (*cf.* Callow, "Hormone Assay," Academic Press Inc., New York, p. 363) with *m*-dinitrobenzene and potassium hydroxide. The oxidations are carried out in the dark with an excess of sodium bismuthate in 5 per cent. acetic acid, when good recoveries of formaldehyde or 17-ketosteroids are obtained. The accuracy and reproducibility of the formaldehyde estimation depends on the rigorous

exclusion of daylight during the oxidation. Substitution of propionic acid for acetic acid in the oxidation medium greatly weakens the effect of light. The procedure is applied to a urinary extract, but with this, purification of the crude non-volatile oxidation product is necessary prior to the estimation of 17-ketosteroids. (See *Brit. Abstr. A III*, 1953, 403.)   I. JONES

132. Microdetermination of polyvinylpyrrolidone in aqueous solution and in body fluids. G. B. Levy and D. Fergus (*Anal. Chem.*, 1953, **25** [9], 1408–1410).—The sample (aq. or saline solution of urine) is diluted with 0·4 M citric acid to adjust the concn. of polyvinylpyrrolidone (I) to between 5 and 30 μg per ml. Ten ml of this solution are treated with 2 ml of 0·006 N iodine solution, the extinction being measured at 5000 A with water as a blank. The (I) content is found by reference to a calibration curve. In the analysis of serum or plasma, a Na tungstate pptn. is performed and the determination of (I) is carried out on the filtrate. Some urines have a strong bleaching action on the (I) - iodine complex, so with these, higher dilutions should be used despite decreased precision at low absorbance. Recoveries are good, being between 92 and 105 per cent.   G. P. COOK

133. Polarographic study of saliva. D. Monnier and Z. Besso (*Anal. Chim. Acta*, 1953, **8** [6], 572–582).—Following earlier procedure (D. Monnier and Z. Besso, *Helv. Chim. Acta*, 1950, **34**, 1842) a polarographic study of diluted buffered saliva in the presence of $CoCl_2$ is reported. Saliva from three individuals is examined and each possesses characteristic features. Certain features of the polarogram are also peculiar to the gland from which the saliva emanates although the results from single glands are not highly reproducible.   W. C. WAKE

134. Microdetermination of copper in biological material. Improved dithizone - polarographic method. D. M. Hubbard and E. C. Spettel (*Anal. Chem.*, 1953, **25** [8], 1245–1247).—An improved dithizone-polarographic method for determining copper in biological material is described. Urine samples of 100 ml or less and 10 g of blood or other tissue samples are prepared for analysis by the standard wet-ashing procedure. Copper is extracted by dithizone and determined polarographically with specificity and sufficient sensitivity.   O. M. JONES

135. Paper chromatography of keto-acids. D. Turnack (*Nature*, 1953, **172**, 355–356).—In order to test the specificity of the Friedemann and Haugen method (*Brit. Abstr. A III*, 1943, 616) for estimation of blood pyruvate, a comparison was made of the chromatograms of blood extracts and of controls of other keto-acids, all run as the hydrazones. The evidence shows clearly that the Friedemann and Haugen method allows separation of pyruvate (with some phenyl pyruvate) from other keto-acids in blood in a more specific way than the method of Cavallini *et al.* (*Brit. Abstr. C*, 1950, 90).
                                    J. L. MALCOLM

136. Influence of formic acid on the hydrolysis of tissue (liver) proteins. S. Gurnani and M. Sahasrabudhe (*Curr. Sci.*, 1953, **22** [9], 269–270).—Pretreatment (bringing to the b.p.) of liver proteins with $H.COOH$ reduces the time required for complete hydrolysis with 2 N HCl. Casein and gelatin are unaffected. Pure amino acids subjected to $H.COOH$ - HCl treatment are only destroyed to the extent of 0 to 6 per cent.   C. G. TAYLOR

Drugs

137. Titration of pharmaceuticals in non-aqueous solvents. C. W. Pifer, E. G. Wollish and M. Schmall (*J. Amer. Pharm. Ass.*, 1953, **42**, 509–521). —A review of various techniques and procedures used in the assay of pharmaceutical compounds by non-aqueous titrations. N. M. WALLER

138. Paper chromatographic identification of some medicaments. A. Wankmüller (*Naturwissenschaften*, 1953, **40**, 57). —Observations on the paper chromatography of sulphonamide derivatives are reported. Diazil [2-(p-aminobenzenesulphonamido)-2:4-dimethylpyrimidine] and Elkosin [6-(p-aminobenzenesulphonamido)-2:4-dimethylpyrimidine] were separated by paper chromatography by use of a mixture of methyl alcohol, amyl alcohol, benzene and water (32:15:45:8). Diazil gave an R_F value of 0·66 and Elkosin 0·73. The spots were identified by spraying the dried strips with 0·1 N HCl containing 0·1 per cent. $NaNO_2$ and then with β-naphthol in alkaline solution. Sulphapyridine and Ladogal could be identified after spraying the strips with 7 per cent. $CuSO_4$ soln. by brown and olive-green spots, respectively. Albucid, sulphanilylurea and Diazil gave faint orange-green spots. Spraying with a concentrated borax solution and examination under u.v. light gave a greenish fluorescence with sulphapyridine and with Ladogal. Ten per cent. aluminium sulphate soln. gave a blue-grey fluorescence with Albucid, steel-blue with sulphapyridine and a green-blue with sulphanilylurea, Diazil and Elkosin. Ladogal itself showed a blue fluorescence without any reagent. Similar investigations on anthraquinone and glycosides indicated that separation could be achieved by a mixture of chloroform, methyl alcohol and water (50 : 32 : 10) and that examination under u.v. light permitted identification of aglycones and glycosides. 1:8-Dioxyanthraquinone gave an R_F value of 0·82, 1:8-dioxy-3-methylanthraquinone, 0·77, and glucose, 0·55. G. W. CAMBRIDGE

139. Qualitative and quantitative analysis of mixtures of sulfonamides. VI. P. L. de Reeder (*Anal. Chim. Acta*, 1593, **9** [2], 140–148). —The discussion is general and concerns the mixtures of pure sulphonamides commonly used therapeutically. Knowledge of the total contents of two or more of C, H, N, or S, enable simultaneous equations to be set up, the solutions of which can lead to the elucidation of the composition of the mixture as the possible components are known. The advantages of the analytical methods available are briefly reviewed. W. C. WAKE

140. Colorimetric determination of dihydro-streptomycin. G. C. Ashton, M. C. Foster and M. Fatherley (*Analyst*, 1953, **78**, 581–585). —A colorimetric method for determination of dihydrostreptomycin is presented. It is based on the reaction between guanido materials and diacetyl, KOH and α-naphthol. The optical density is measured at 525 mμ precisely 40 min. after reaction, and the potency of the sample solution is determined by means of a calibration graph prepared from standard dihydrostreptomycin. The diacetyl reaction is given by streptomycin and other guanido derivatives. Penicillin does not interfere. A. O. JONES

141. Colorimetric determination of erythromycin. J. H. Ford, G. C. Prescott, J. W. Hinman and E. L. Caron (*Anal. Chem.*, 1953, **25** [8], 1195–1197). —An erythromycin solution, containing 50 μg per ml, is prepared in phosphate buffer (pH 7·0) as solvent. Five ml of 27 N H_2SO_4 are added to 5 ml of this solution and the absorbancy is measured at 485 mμ after 30 min. The erythromycin content of the sample is found by reference to a suitable calibration graph. For the analysis of brew, 20 ml of the suitably diluted brew is extracted with amyl acetate, 10 ml of the solvent layer is extracted with 0·1 N HCl, and the determination of erythromycin is carried out on the aq. phase. The results are reproducible to ± 1 per cent. on aq. solutions and ± 3 per cent. on brews. Degradation products of erythromycin interfere. G. P. COOK

142. Applications of flame photometry to the analysis of alkalis and calcium in saline solutions for infusion. Per Lundgren (*J. Pharm. Pharmacol.*, 1953, **5** [8], 511–527). —Flame photometry will give the clinician in a very short space of time data on the sodium and potassium concentration in urine, plasma etc., and this method is assuming great importance amongst the practical sciences. Because the theoretical connection between the concentration of the element in the flame and the intensity of its emission is not yet elucidated, quantitative flame photometric analysis is based upon an empirical comparison of the test solution with standards. The investigations described in this paper were carried out in order to serve as a basis for the application of this method to the routine control of saline solutions in the laboratory; the procedures outlined were applied to about 100 solutions of various compositions. The method can be applied to various substances in pharmaceutical analytical work. J. CALEY

Food

143. Determination of D-glucose in corn syrups. Use of glucose dehydrogenase. R. L. Whistler, L. Hough and J. W. Hylin (*Anal. Chem.*, 1953, **25** [8], 1215–1216). —Glucose dehydrogenase catalyses the oxidation of β-D-glucose to D-gluconolactone and H_2O_2. A solution containing $\simeq$ 20 mg of D-glucose is mixed with the enzyme solution, and O is passed through for 4 hours at 30° C. The D-gluconolactone produced is determined by titration with 0·01 N NaOH using phenolphthalein as indicator, at a temp. of 50° to 60° C. Agreement with other methods is good. G. P. COOK

144. Unneutralised hydrolysates in paper chromatography of sugars. B. D. E. Gaillard (*Nature*, 1953, **171**, 1160). —Good separation of acidified sugar mixtures and of the sugars in unneutralised polysaccharide acid hydrolysates was obtained by use of benzene – butanol – pyridine – water (1:5:3:3) as solvent. The sprays used were aniline phosphate or phthalate. H. G. BEVAN

145. Potentiometric iodine titration of branched starch fractions. J. F. Foster and Richard L. Smith (*Iowa State Coll. J. Sci.*, 1953, **27**, 467–477). —A routine potentiometric method for determination of combined I in starch derivative - iodine complexes is described.

A 40 to 500-mg carbohydrate sample is dissolved in 5 ml of N aq. KOH and the solution brought to pH 6·3 with 0·5 N HCl. 0·5 N aq. KI is added so that on dilution to 100 ml, the solution is 0·05 to 0·30 N with respect to KI. This solution is titrated potentiometrically to null-point with 0·016 N I soln. in aq. KI and KCl at the same normality as the titrated solution, with reference to a cell containing

a solution prepared as above without the starch derivative. Titration curves are presented for potato, corn and tapioca starch, amylose and amylopectin and for waxy maize starch and glycogen; the results are discussed in relation to the mechanism of complex formation with I.   D. A. PANTONY

146. Accuracy of quantitative paper chromatography in amino acid analysis—Addendum. R. C. Salander, M. Piano and A. R. Patton (*Anal. Chem.*, 1953, **25** [8], 1252–1253).—An experimental study of the accuracy of Patton and Chism's quantitative method for paper chromatography of amino acids (*Anal. Chem.*, 1951, **23**, 1683). Some previously unpublished details of procedure are reported.
                                                        O. M. JONES

147. Paper chromatography in aqueous phase for the study of dinitrophenyl (DNP) amino acids and peptides. M. Rovery and C. Fabre (*Bull. Soc. Chim. Biol.*, *Paris*, 1953, **35** [5–6], 541–546).—DNP amino acids and DNP peptides can be chromatographed on paper by use of an aqueous mobile phase of citrate and HCl at pH 6·2. The order of spots is completely different from that obtained with organic solvents. Not all DNP amino acids can be identified by examination of a single chromatogram, but the groups are segregated and can be easily separated further by another mobile phase. Several identifications of DNP amino acids made by means of the classical silica gel columns have been controlled by this new technique. Agreement is excellent save for the band of DNP-phenylalanine from α-chymotrypsin which conceals DNP-*iso*leucyl-valine the stability of which is exceptionally great. This peptide derives from the *iso*leucine chain of chymotrypsin.   J. G. HEATHCOTE

148. Application of ion exchanger chromatography to the study of the amino-acid composition of food-stuffs. E. Schram, J. P. Dustin, S. Moore and E. J. Bigwood (*Anal. Chim. Acta*, 1953, **9** [2], 149–162).—It is important to examine a hydrolysate prepared from the foodstuff rather than a protein extract. The possible interference from carbohydrates is investigated by the use of tapioca starch as the source of the amino acids. The proteins in the sample are hydrolysed by heating under reflux for 24 hr. with 6 *N* HCl. Chromatography is carried out on an ion-exchange resin, Dowex-50, and an automatic fractionation device is used to divide the effluent. Ninhydrin reagent permits the course of the elution to be followed. Cystine and cysteine are unstable in the presence of carbohydrate whilst methionine is oxidised under the conditions of the separation. Tryptophan cannot be determined by this method.   W. C. WAKE

149. Modification of the van Gulik method for determination of fat in soft cheese. A. Pinsky and A. Grünpeter (*Analyst*, 1953, **78**, 621–622).—The van Gulik method for determination of the fat content of cheese was found unsatisfactory in the examination of a soft skim milk cheese produced in Israel from milk powder to which rendered fat or margarine is added to adjust the fat content of the cheese to 9 per cent. By the standard procedure the fat separated imperfectly on centrifuging and contained opaque particles of non-fatty matter. When the sp.gr. of the H_2SO_4 used was raised to 1·70, other details of the method remaining unchanged, the cheese mass was dissolved in 1 hour. Final separation was clear and results agreed with those of a gravimetric method.   A. O. JONES

150. Thiobarbituric acid test for butter-fat oxidpantothe**tion.** D. A. Biggs and L. R. Bryant (*Canad.* 1 hr. with Technol.*, 1953, **31** [6], 138–145).—The method H$_2$SO$_4$ an Dunkley and Jennings (*J. Dairy Sci.*, 1951, **34**, 106 the hydr 1069) has been improved by (*a*) using an emulsify2 : 7-naph agent to ensure adequate contact between t an an ice separate phases of substrate and reagents; (*b*) fixi boiling w the pH of reaction at 1·6; (*c*) performing t cooled in reaction in a strictly inert atmosphere; (*d*) correcti and the the values obtained from milk products for t465 mμ colour produced by reaction with lactose, whi reagents. interferes. *Method*: A weighed portion of sample in vitar placed in a three-necked flask, through which oxyge chromat free nitrogen has been passed for 15 min., equippe column; with a condenser, mercury seal and stopper; the removed 75 ml of thiobarbituric acid reagent (0·025 *M* other su *M* H$_3$PO$_4$) and approximately 5 ml of 10 per cen fere. T alkyl-dimethylbenzylammonium chloride (Rocca biologica are added. The reaction mixture is heated in a ba of boiling water. After 1 hour, a 10-ml aliquot **155. I** withdrawn and extracted with 10 ml of a mixtu riboflavi of 1 part of pyridine to 2 parts of amyl alcoho assay After centrifugation to clarify the extract, th Clarke (optical density of the solvent is measured at 535 m study s in a Beckman (model B) spectrophotometer an reliabili the results expressed as optical density units. of ribof
                                        L. G. L. UNSTEAD-JOSS be impr
                                                          potency
151. The chromatographic examination of permi the glue **sible food dyes.** J. R. A. Anderson and E. C. Marti amount (*Anal. Chim. Acta*, 1953, **8** [6], 530–537).—The dye and soc stuffs are 36 of those permitted in New South Wale Australia. An ascending technique was used wit four different developing agents used individually i one dimension only. R_F values are given fo **156.** aqueous solutions and for solutions obtained b plexon extracting the dyestuffs with butanol saturate T. B. S with 2 *N* HCl. Some results obtained by extractin sky (*J* commercial jelly crystals with this solvent ar The be quoted. The best developing agent is phenol toward water-formic acid. The R_F value of a monaz discuss dyestuff is a function of the number of –SO$_3$O interfe groups.   W. C. WAKE ness a

152. Paper-chromatographic separation of B 157. group vitamins. R. Radhakrishnamurty and P. S and su Sarma (*Curr. Sci.*, 1953, **22**, 209–210).—Most of th **25** [8] B-vitamins, *viz.*, thiamine hydrochloride, riboflavin as 0·0 choline chloride, inositol, nicotinic acid, nicotin determ amide, B$_{12}$, Ca pantothenate, biotin, pyridoxine Halle hydrochloride and *p*-aminobenzoic acid, have been remove successfully separated from a mixture, and identified Resul by means of ascending chromatography, specifi adsor chemical reagents being used for the individual amou vitamins. This method and also radial chromato tion o graphy have been used for the identification o direct various constituents in commercial vitamin tablets recov
                                                      A. STORFER

153. Colorimetric determination of thiamine i pharmaceutical products. F. J. Bandelin and J. V 15 Tuschhoff (*Anal. Chem.*, 1953, **25** [8], 1198–1200).— cide-A solution containing 2 to 5 mg of thiamine i photo acetate buffer at pH 4·5 is treated with 5 ml of 2 Edw per cent. NH$_4$ reineckate in methanol, and filtered The after 30 min. standing. The ppt. is washed 3 times and with dil. reineckate solution and then dissolved in hexa 10 ml of acetone. The absorbancy is measured at estim 525 mμ. The accuracy and reproducibility of the then method are within 2 per cent. Certain heterocyclic abso amines and quaternary NH$_4$ salts interfere. blan
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154. Colorimetric determination of calcium panto- 50 t **thenate.** C. R. Szalkowski and J. H. Davidson, jun. Prec (*Anal. Chem.*, 1953, **25** [8], 1192–1195).—Calcium

pantothenate (25 mg) is heated under reflux for 1 hr. with 40 ml of water and 5 ml of 50 per cent. H_2SO_4 and then diluted to 100 ml. Aliquots of the hydrolysed solution are treated with 10 ml of 2 : 7-naphthalenediol (1 mg per ml in conc. H_2SO_4) in an ice bath, and then immersed in a bath of boiling water for 30 min. Each solution is once more cooled in ice, 15 ml of 50 per cent. H_2SO_4 are added and the absorbancy is measured after 30 min. at 465 mμ against a blank prepared from the same reagents. In the determination of pantothenate in vitamin mixtures, riboflavin is removed by chromatography of the aq. sample on a Florisil column; ascorbic acid, glucose and lactose are removed by $Ca(OH)_2$ and $CuSO_4$ treatment. No other substances found in vitamin mixtures interfere. There is good agreement with the microbiological method. G. P. Cook

Sanitation

155. Media used for microbiological assay of riboflavin using *Lactobacillus casei*. Method of assay and confidence limits attainable. M. F. Clarke (*Anal. Chem.*, 1953, **25** [8], 1247–1252).—A study shows that the susceptibility to tests of reliability of media commonly used in the estimation of riboflavin potency by microbiological assays can be improved, and the confidence limits of estimated potency can be more simply calculated by increasing the glucose content to 3 per cent. and changing the amounts of yeast preparations, ammonium sulphate, and sodium acetate. O. M. Jones

Sanitation

156. Behaviour of certain cations in the complexone method of determining hardness of water. T. B. Styunkel, E. M. Yakimets and D. A. Savinovsky (*J. Anal. Chem., U.S.S.R.*, 1953, **8**, 163–167).—The behaviour of Ca, Mg, Zn, Mn, Cu, Al, and Fe towards Trilon B in presence of various indicators is discussed and methods of avoiding the effects of interfering ions in the determination of water hardness are described. G. S. Smith

157. Microdetermination of DDT in river water and suspended solids. B. Berck (*Anal. Chem.*, 1953, **25** [8], 1253–1256).—DDT in river water (as low as 0·003 p.p.m. of DDT) and in suspended solids was determined by solvent extraction and the Schechter-Haller procedure. Interferences in the extract were removed by Davidow's chromatographic method. Results showed that solids suspended in river water adsorbed DDT. In samples containing sub-optimal amounts of DDT, for which regular solvent extraction of water was not feasible, DDT was determined directly from the suspended solids fraction. Mean recoveries vary greatly. O. M. Jones

158. Determination of Methoxychlor on insecticide-treated paperboard by ultra violet spectrophotometry. E. C. Jennings, jun. and David G. Edwards (*Anal. Chem.*, 1953, **25** [8], 1179–1182).—The treated board is cut into strips of suitable size and extracted in a Soxhlet extractor with *cyclo*hexane, the time of extraction varying with the estimated Methoxychlor content. The extract is then concentrated using a Vigreux column, and the absorbancy at 230 mμ is compared with that of a blank extraction. Background interference is corrected for by the double impurity index method. Recoveries of 92·6 ± 2·2 per cent. are attained for 50 to 1000 mg per sq. ft. additions to paper board. Precision is ± 5 per cent. G. P. Cook

Agriculture

159. Microdetermination of N-1-naphthylphthalamic acid residues in plant tissues. Allen E. Smith and G. M. Stone (*Anal. Chem.*, 1953, **25** [9], 1397–1399).—The N-1-naphthylphthalamic acid (**I**) is converted to 1-naphthylamine and Na phthalate by hydrolysis with NaOH. The 1-naphthylamine is separated by steam distillation and then coupled with diazotised sulphanilic acid giving a red azo dye, which is determined colorimetrically. The λ used for measurement is 534 mμ, and a reagent blank is used as reference solution. The diazonium coupling reaction is carried out in 20 per cent. acetic acid and the formation of the dye is complete in ≃ 30 min., the colour being stable for 2 hr. The limit of detection of (**I**) is 2 μg, and 80 to 100 per cent. recoveries are attained in the 5-μg range. The same method is applicable to the determination of the salts, esters and imide of (**I**). G. P. Cook

160. Detection of hexachlorocyclohexane in vegetables. J. F. Reith (*Chem. Weekbl.*, 1953, **49**, 689–692).—In a simplified version of Schecter and Hornstein's method (*Anal. Chem.*, 1952, **24**, 544), the apparatus (reduced in size) consists of a spherical 15 ml-flask fitted with a reflux condenser (total height ≃ 20 cm), the inner tube of the condenser being bent round at the top to connect with a U-tube, one limb of which carries a bulb and the other a widened tube (≃ 7 cm long and 12 mm in diameter) containing glass beads (nitration chamber), which is surmounted by a soda-lime tube; ground-glass joints are used. The condenser jacket is filled with water at 10° to 15° C (instead of at 80° C). Ether (100 + 50 + 50 ml) is used for extracting (by decantation) hexachlorocyclohexane (**I**) from the finely ground material (50 g). Concentration of the extract to ≃ 10 ml is carried out during about 20 min., and the final 10 ml (with rinsings of ether) are allowed to evaporate in a dish at or near room temp. The residue is transferred to the flask (containing 1 g of Zn dust and 2 g of malonic acid) with 7 ml of conc. acetic acid, and the beads in the nitration chamber are charged with 3 ml of a mixture of conc. H_2SO_4 and fuming HNO_3 (1 + 1). The bulk of the benzene (formed by reduction of the **I**) is evaporated through the condenser and into the nitration chamber by boiling (micro-burner) the reaction mixture vigorously for 1 hour. The nitration mixture, diluted with 15 ml of water and cooled, is transferred (rinsing with a few ml of water) to a separating funnel and is extracted with ether (15 ml). The ether layer is shaken out with water (15 ml), then with 0·5 N NaOH, 0·1 N NaOH, and water (5 ml of each). After addition of 5 drops of liquid paraffin (in order to prevent loss of **I** by evaporation), the ether is slowly evaporated in a dish, and the residue, dissolved in 3 ml of methyl ethyl ketone, is shaken for 1 min. in a glass-stoppered test tube with 1 ml of 50 per cent. KOH. A violet coloration in the upper layer (produced within 5 min.) indicates the presence of (**I**) in the sample. A quantitative determination is possible with the use of a colour standard (60 ml of 0·25 M $CuSO_4$ and 40 ml of 0·01 N $KMnO_4$. The reaction is sensitive to 1 p.p.m. of (**I**). Naturally occurring substances or other insecticides (DDT, Aldrin, Dieldrin, Chlordane, or Parathion) do not interfere with the test. P. S. Arup

161. Analysis of commercial allethrin. Determination of allethrin content by reaction with ethylenediamine. J. N. Hogsett, H. W. Kacy and

J. B. Johnson (*Anal. Chem.*, 1953, **25** [8], 1207–1211).—Allethrin (DL-2-allyl-4-hydroxy-3-methyl-2-*cyclo*penten-1-one) is reacted with ethylenediamine to form chrysanthemum monocarboxylic acid, which is determined by titration with standard sodium methoxide in pyridine solution with thymolphthalein as indicator. Chrysanthemum monocarboxylic acid, its anhydride and acid chloride also react quantitively and are determined separately, the corrections applied giving the true allethrin assay. The acid chloride is determined by reaction with methanol and titration of the HCl formed with methanolic KOH and by use of dimethyl yellow-methylene blue indicator; the acid plus acid chloride is determined by titration with NaOH to α-naphtholbenzein indicator. Acid chloride plus anhydride is determined after reaction with excess of morpholine by titration of the excess with methanolic HCl and dimethyl yellow-methylene blue indicator. The results agree well with the Peet-Grady biological method.   G. P. COOK

162. **Colorimetric determination of pyrethrins, allethrin and furethrin.** C. Cueto and W. E. Dale. (*Anal. Chem.*, 1953, **25** [9], 1367–1369).—When the sample consists of paper or cloth bags, impregnated with the insecticide, alcoholic solutions of the insecticides are prepared by Soxhlet extraction. A 1 per cent. solution of Na₂S is added and the solution is heated at 70° ± 5° C for exactly 15 min., the absorbancy of the orange to red colour produced being measured after 10 min. at 540 mμ. The amount of the insecticide is calculated by reference to a suitable calibration curve, Beer's law being obeyed for 0·5 to 10·0 mg of pyrethrins, allethrin and furethrin. The average experimental error is ≃ 4 per cent. and the method can only be applied if it is known what insecticide is present in the sample.   G. P. COOK

See also Abstracts 49, 70, 109, 116, 170.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

163. **Paper chromatography.** M. Milletti (*Ann. Chim., Roma*, 1953, **43**, 338–344).—A technique for paper chromatography by the ascending method is described; it makes use of paper strips constricted near the bottom and suspended in test tubes. The method is applied to the separation of mixtures of amino acids, the chromatograms being developed either with ninhydrin, or by exposure to the vapour of salicylaldehyde to give fluorescent or coloured azomethines.   J. K. LANDQUIST

164. **New type of qualitative chromatographic paper column.** C. E. Danielsson (*Ark. Kemi*, 1953, **5**, 173–175).—The large-scale chromatographic method of separation described and illustrated depends on use of a rolled-paper column, 30-cm high and of 10 cm outer diameter, the central hole (1 cm diameter) being filled with paraffin wax. Seven concentric filter-paper rings (inner radius, 1·5 cm, outer 4 cm) are saturated with a known amount of the test solution and are placed on the top of the column, which is saturated with the solvent. A similar paper column, but 10 cm high only, is placed on the rings on the top of the large column, and is kept saturated with solvent by means of a simple device. Straight fronts are obtained because the speed of elution at 90° to the axis of the 30-cm column is constant. The column is finally cut

along its vertical axis and the ninhydrin reaction applied at different distances from the axis so as to fix the position of the separated components. An effective qual. separation of valine, glycine, and alanine (25 mg of each) dissolved in a mixture of 70 per cent. ethanol and 5 per cent. acetic acid is attained.   W. J. BAKER

165. **A simple device to keep cylinders of paper inside glass cylinders for paper chromatography.** G. S. Siddappa and B. S. Bhatia (*Curr. Sci.*, 1953, **22** [8], 239).—Filter-paper, spotted with test soln, is made into a cylinder by sewing the edges together with thread, and has a diameter ½ to ¾ in. less and height approx. 1 in. less than the glass cylinder in which it stands. A filter-paper cone is placed with its tip inside the paper cylinder and is of such a size that its rim is flush with the top of the glass cylinder. The glass cylinder contains the solvent and is covered with a ground-glass plate during development.   E. C. BUTTERWORTH

166. **Effect of temperature on movement of chromatographic zone.** Lie Tien Chang (*Anal. Chem.*, 1953, **25** [8], 1235–1237).—An experimental study is described of the effect of temperature on the R_F value, within − 50° to 200° C. Results are reported for experiments with azobenzene, *p*-hydroxyazobenzene, aminophenylazo-2-naphthol, 1 : 4-dibutylaminoanthraquinone, or *p*-aminoazobenzene as adsorptive, silicic acid as adsorbent, and diphenyl ether, *n*-butyl ether, or *iso*propyl ether as solvent.   O. M. JONES

167. **Filter paper disk chromatography.** K. V. Giri (*Nature*, 1953, **171**, 1159).—The technique has been reported previously (*Brit. Abstr. C*, 1952, 346, etc.). Further experience shows that with 35-cm filter-papers and mixtures of *n*-butyl alcohol, acetic acid and water (40:10:50), pyridine and water (80:20), or pyridine, amyl acetate and water as solvents all amino-acids of protein hydrolysates can be separated.   H. G. BEVAN

168. **Investigations on circular paper chromatography.** H. C. Chakrabortty and D. P. Burma (*Curr. Sci.*, 1953, **22** [8], 238–239).—The R_F values of amino-acids by circular paper chromatography are higher than when uni-dimensional methods are used. This is due to the orthogonal distribution of the solvent.   E. C. BUTTERWORTH

169. **Quantitative paper chromatography of traces of metal with the aid of radioactive hydrogen sulphide.** P. C. van Erkelens (*Nature*, 1953, **172**, 357–358).—Samples are ashed, the C destroyed with NH₄NO₃ and H₂O₂, and the residue is dissolved in HCl; the solution is chromatographed on carefully washed paper. A variety of novel solvents is recommended. The dried paper is conditioned over aq. NH₃, and put into a bell-jar with aq. NH₃, N₂H₄, and Zn³⁵S under N; acid is added and the apparatus left in the dark for 10 min. The method is applicable to any metal, is cheap, can be carried out on a large scale, and has a sensitivity comparable with that of spectrography.   H. P. PAGET

170. **A simple apparatus for paper electrophoresis.** S. S. Rao (*Curr. Sci.*, 1953, **22** [9], 274–275).—A description is given of an apparatus for the separation of proteins suitable for two-dimensional work with micro-amounts of solutions (blood serum).
                                    C. G. TAYLOR

171. **Method of analysing flow of granular materials such as sand.** J. Gittus (*Nature*, 1953, **172**, 364).—It is suggested that the flow characteristics

of a granular material are determined by the relative values of the energies required to coagulate and to disperse the grains. For a material falling through a given distance on to a given sieve, the amount passing through the sieve is then inversely proportional to the energy required for dispersion; the prediction is modified for samples of sand bonded with different clay - water mixtures. The principle is useful in the analysis of many flow problems.   H. P. PAGET

172. Calibration of air permeability particle sizers. B. Dubrow (*Anal. Chem.*, 1953, **25** [8], 1242–1244).— The subsieve sizer, an air permeability apparatus for determining the average particle size of fine powders, can be calibrated by the two procedures described with the aid of fritted-glass diffusion tubes as standards. In one, the air flow resistance of the glass diffusion tube is measured by a manometer. The other utilises a wet-test meter to determine the air conductance through both the diffusion tube and the capillary flowmeters. Results are reported for glass diffusion tubes having porosities corresponding to nominal pore sizes from 0·9 to 60 microns.
    O. M. JONES

173. Determination of size distribution of macropores in porous materials. Macroporosity of coke by gas flow method. S. Ergun and Jack Owen (*Anal. Chem.*, 1953, **25** [8], 1222–1226).—A method is reported for the analysis of size distribution and estimation of surface areas of macropores in porous materials from the measurements of aerodynamic densities of fractions of various sizes by the gas flow method. Some results for metallurgical cokes are presented and discussed. Calculated surface areas agree well with data reported in the literature.
    O. M. JONES

174. A differential capacitance manometer. I. G. Baxter (*J. Sci. Instrum.*, 1953, **30** [10], 358–360).— The manometer has been designed for use in a blood-flow meter. The pressure difference is applied across a metal diaphragm, the centre of which is linked to a lever passing through a flexible metal membrane. A capacitance pick-up is mounted at the other end of the lever. Pressures within the range 0·03 to 15 mm of Hg can be measured.
    G. SKIRROW

175. Microgasometric procedure. H. G. Heal (*Nature*, 1953, **172**, 30).—A simple method is described for the determination of small volumes of gas (several μl) evolved when a 1 to 2-mg crystal dissolves in water. The crystal is placed on a microscope slide and water is added beneath the cover slip; the diameter of the bubble of gas formed is measured by means of a travelling microscope. Full details are given.   B. VINEY

176. Plastic stirrer blades. V. R. Shellman and B. J. Magerlein (*Anal. Chem.*, 1953, **25** [8], 1285). —Three Teflon stirrer blades are illustrated and their construction is outlined. One is patterned after the Trubore glass stirrer blade of Ace Glass, Inc. and the other two are reminiscent of the wide stirrer described by Hershberg (*Ind. Eng. Chem., Anal. Ed.*, 1936, **8**, 313). These stirrer blades are superior to their glass or wire counterparts in that, they are easily cleaned, do not scratch and are nearly indestructible.   O. M. JONES

177 A laboratory stirrer for viscous liquids. M. D. Ashton (*J. Sci. Instrum.*, 1953, **30** [10], 388).— The stirrer consists of an approx. 1-inch diam. rod 6 inches long, having a helical insert fitting into a

hole 3 inches deep bored at one end. On rotating the rod at speeds of 2000 to 3000 r.p.m., the liquid is forced up the cavity and ejected as a fine spray from a number of small radial holes at the upper end of the cavity.   G. SKIRROW

178. Simply constructed laboratory valve. James Walker (*Chem. & Ind.*, 1953, [34], 888).—A laboratory excess pressure control valve is described for use with low-pressure hydrogenation equipment. It consists of a 1-inch piece of 5 mm bore glass tube fused to the sealed bottom of a wide tube with a side-arm. A $1\frac{1}{4}$ to $1\frac{1}{2}$-inches spiral spring (uncompressed) encircles the inner tube and holds a hemispherical (Holdtite) tap washer against the flared-out end of a 7 to 8 mm bore tube held in a rubber stopper and connected with the gas-vacuum line. The outer tube is filled with water to a point $\frac{3}{4}$ inch above end of the gas inlet tube.   D. BAILEY

179. Glass to metal seal through aluminium using the Al - Fin bond. R. Aves (*J. Sci. Instrum.*, 1953, **30** [10], 388).—The seal consists in the sequence Al, Al - Fin bond, Nilo-K, Kodial. The seal is useful for introducing electrodes to a metal container via a glass seal.   G. SKIRROW

180. Centrifuge type of ultra-filtration apparatus. T. Y. Toribara (*Anal. Chem.*, 1953, **25** [8], 1286).— An all-glass one-piece centrifuge for ultra-filtration is illustrated and described. One end of a borosilicate glass tube, 25 mm in diameter, with coarse fritted disc is sealed off. The filtrate is removed and the same atmosphere maintained above and below the fritted disc by a 6-mm glass tube sealed off at an angle just below the disc. A cellophane dialysing tube holds the solution to be filtered. Dog blood plasma can be filtered at speeds up to 2000 r.p.m., and, without the cellophane bag, precipitates can be dried by centrifugation.
    O. M. JONES

181. Ultrafiltration in the centrifuge. H. Metzner (*Naturwissenschaften*, 1953, **40**, 388–389).—Originally designed for separation of homogeneous tissue from cellular fragments, a new centrifuge (described) is used for ultrafiltration, such as the separation of microsome and nuclear fragments from cells and for the concentration of dil. protein solutions. The centrifuge can be sterilised.   R. J. MAGEE

182. Precise speedy pipette. C. H. Whitnah (*Anal. Chem.*, 1953, **25** [8], 1285–1286).—The pipette described and illustrated measures pyridine sulphate dibromide reagent for iodine numbers by a modified Rosenmund and Kuhnhenn method. Density and concentration can be considered constant during the two blank and four duplicate measurements. The pipette may be considered as a Koch burette modified by the replacement of the burette tube by a tube of about 8-mm int. diam. and 5-ml capacity with about 1·5 mm of capillary at each end. Scratches on a mirror behind this tube replace upper and lower graduations on the pipette. The upper capillary is sealed to a water manometer and an air reservoir graduated from the top downwards. It can be partly filled with water from a levelling bulb with a stopcock. When the air volume and pressure in the reservoir are properly adjusted by a stopcock near the top, increased pressure stops flow of reagent into the measuring tube just above the upper scratch; decreased pressure stops flow just below the lower scratch. Tabulated data indicate precision of water delivery.
    O. M. JONES

183. Overflow micro-pipette for rapid and accurate measurement of small volumes of liquid. H. A. Sissingh (*Chem. Weekbl.*, 1953, **49**, 722–723).—The measuring tube of the required vol. (0·5 to 5 ml), $\simeq$ 15 cm long, is drawn out to 0·8 to 1·5 mm bore at each end, the upper end being bent at $\simeq$ 45° and fitted into a wider tube (rubber stopper), which is closed by a rubber stopper at the top. The wider tube has two short side-tubes—the upper one open and the lower one closed—of suitable shape for fitting tightly into perforations in a piece of rubber tubing, which is closed at either end and is about as long as the wide tube. By pressing and releasing the rubber tube, the liquid is sucked into the measuring tube, and is delivered by pressure. The inside of the apparatus is coated with Desicote. Directions are given for making the measuring tube of the desired capacity. Deliveries are accurate to 0·1 per cent.   P. S. ARUP

184. Macromicroburettte. K. F. Körner (*Z. anal. Chem.*, 1953, **139** [2], 99–100).—An autofilling macromicroburette is described; details of its construction and a diagram are included. G. P. COOK

185. Apparatus for simplifying titrations in a controlled atmosphere. R. H. Prince (*Analyst*, 1953, **78**, 607–613).—Construction and use of apparatus for titration in a controlled atmosphere are described in detail. The apparatus is especially suited to Fischer determinations of water; such a determination of moisture in transformer oil is described.   A. O. JONES

186. An apparatus for the microanalysis of gas mixtures. M. J. Marshall and G. Constabaris (*Canad. J. Chem.*, 1953, **31** [9], 842–848).—An apparatus is described for the analysis of gas volumes as small as 5 cu. mm at N.T.P. in the dry way. Two applications are presented, one for CO_2, the other for H by diffusion through Pd in the presence of CO or CH_4. The preparation of an absorption reagent for CO_2 consisting of 2·6 per cent. LiH, 23·6 per cent NaOH and 73·8 per cent. KOH is described.
                              D. LIFF

187. A robust air-flow meter for field use. D. S. M. Phillips (*J. Sci. Instrum.*, 1953, **30** [10], 382–383).—The air flows through a series of small orifices arranged in a semi-circle in a smooth metal plate. Provision is made for uncovering the orifices one at a time until the pressure in the system falls to a specified level. The number of orifices uncovered then gives a measure of the rate of flow of air into the system.   G. SKIRROW

188. Semi-micro distillation columns. M. Boivin (*Chim. Anal.*, 1953, **35** [8], 182–187).—Detailed drawings are given for a spinning band distillation apparatus having a 3·3-mm × 0·25-mm Ni - Cr ribbon about 550 mm long. The band is attached to the motor by a Ni - Cr wire led through a glass capillary and has a max. speed of 5000 r.p.m. The effect of rate of rotation on the separation is illustrated by reference to the separation of a mixture of benzene and toluene. There is a marked separation of pure benzene when the band is spinning at 2000 r.p.m., and separation is improved slightly by increasing the speed to 5000 r.p.m. By contrast, no pure benzene is distilled over when the band is stationary. Other separations are illustrated together with data on reflux rate and take-off ratio.   W. C. WAKE

189. Calibration of a Stormer viscosimeter with special reference to turbulent flow conditions. P. J. van der Walt and A. M. Fourie (*J. S. Afr. Chem. Inst.*, 1953, **6**, 36–47).—A relationship between the coefficient of resistance and the Reynolds No. can be determined for a Stormer viscosimeter for a wide range of η and types of rotor. The principles described may be applicable to other types of viscosimeters.   J. DAVIS

190. New method of measuring surface tension of liquids using the vertical liquid jet. T. Ikeda (*Bull. Chem. Soc. Japan*, 1953, **26** [6], 352).—An approximate equation has been derived, $V_e = \pi r^{3/2} (\sqrt{2\gamma/\rho})$, where V_e is the starting critical flow rate necessary to change the tripping flow of the vertical jet into the laminar flow, r is the inner radius of the nozzle, γ the surface tension and ρ the sp.gr. of the liquid. The formula is accurate for $r < 0.05$ cm. Results for several liquids are reported. Water, $CHCl_3$ and CCl_4 gave anomalous figures.   C. D. LAFFERTY

191. A simple method of leak finding using a Pirani gauge. R. N. Bloomer and M. E. Haine (*J. Sci. Instrum.*, 1953, **30** [10], 385–386).—Application of a probe vapour such as ether or butane to a leak in a continuously evacuated system results in an indication of pressure increase by a Pirani gauge connected on the high vac. side of the vapour pump.   G. SKIRROW

192. A simple direct-reading friction meter. V. E. Gough (*J. Sci. Instrum.*, 1953, **30** [10], 345–349).—A platform carried by four identical levers each with a counterweight is in neutral equilibrium. One of the two surfaces under test is that of the platform. The coefficient of friction is the tangent of the angle between the levers and the vertical when the other test surface is dragged along the platform.
                              G. SKIRROW

Optical

193. Microphotometer for X-ray analysis of crystals. L. Cavalca (*Ric. Sci.*, 1953, **23** [9], 1586–1592).—The microphotometer of Bezzi (*Gazz. Chim. Ital.*, 1940, **70**, 160) is modified so that the photo-electric cell is fixed and the projection of the photograph is movable. The instrument is suitable for measuring the diffraction intensities of Wiebenga and Smits photographs as well as those of single crystal and Debye photographs.   E. HAYES

194. Source unit for Raman spectroscopy. J. W. Kemp, J. L. Jones and R. W. Durkee (*J. Opt. Soc. Amer.*, 1952, **42**, 811–814).—A "Toronto" type of helical lamp, containing a four-turn helix of 25-mm Pyrex-glass tubing and a pair of Hg pools as electrodes serves as a source for Raman spectroscopy. Ratios obtained of the 4347 A and 4339 A Hg lines with background at 4358 A are found to be of the order of 0·08 to 0·0005. Comparative data for several lamps used in Raman spectroscopy indicate that the lamp compares favourably with the best 10-amp. lamp. Details of the power supply unit are given.   E. L. SEYMOUR

195. Determination of mineral constituents of rocks by infra red spectroscopy. John M. Hunt and Daniel S. Turner (*Anal. Chem.*, 1953, **25** [8], 1169–1174).—The rock sample is pulverised with a micronizer, so that 90 per cent. of it consists of particles smaller than 5μ. A few mg of the powder are placed on a standard NaCl window, and a few

drops of *isopropyl* alcohol added to make a paste, which is smoothed out leaving a powder film when the alcohol has evaporated. The edge of the film is cleaned off to a premarked position on the window and an infra-red spectrum obtained in the usual manner, the mineral constituents being determined by reference to suitable standards. Quant. analyses to within 10 per cent. can be obtained for minerals such as quartz, kaolinite, orthoclase, calcite and dolomite. Errors are due to non-uniformity of the film and scattering of the radiation.   G. P. COOK

196. Analytical applications of infra-red spectroscopy in the gas industry. J. Garach (*Chim. Anal.*, 1953, **35** [9], 207–218).—The paper gives a general review of principles and apparatus and concludes with examples. Those discussed include the quantitative determination of C_3H_8 and C_3H_6, and identification of hydrocarbons and coal-tar constituents. A list of reference spectrograms concludes the paper.   W. C. WAKE

197. A water-jacketed cell-holder for use with the Unicam SP 500 spectrophotometer. H. Campbell and J. A. Simpson (*Chem. & Ind.*, 1953, [34], 887–888).—An apparatus is described which allows water at constant temp. to be circulated about two standard cells of a Unicam SP500 spectrophotometer. The water jacket and cells, which are in contact to ensure good heat transfer, are moved together as a unit during the normal operation of the spectrophotometer. Flooding of the cell compartment is avoided by the use of sliding water inlet and outlet tubes. The temperature variation in the cells is < ± 0·05° C. The construction of the apparatus is described and illustrated.
D. BAILEY

198. A projector-comparator for examining spectrographic plates. J. C. North (*J. Sci. Instrum.*, 1953, **30** [10], 366–369).—The instrument described permits comparison of two spectrographic plates. Magnification of × 20 is given, and provision is made for independent manipulation of each plate.
G. SKIRROW

199. Semiquantitative spectrographic method for analysis of minerals, rocks and ores. C. L. Waring and C. S. Annell (*Anal. Chem.*, 1953, **25** [8], 1174–1179).—The sample is mixed with graphite and placed in a prepared electrode, which is subjected to a direct current arc. A grating spectrograph gives a range from 2250 to 4750 Å, which is covered on 2 adjacent plates. The unknown elements are estimated by visual comparison from standards, which are prepared for each element, the content decreasing from 10 to 10^{-4} per cent. This method enables 68 elements to be determined on exposure of a 10-mg sample to 1 arc, each element being reported in a concn. range. Ninety-two per cent. of the determinations are in agreement with chemical results, the remaining 8 per cent. agree within the concn. range. Details of the preparation of the standards are given, including a table of the wavelengths used for the identification of the elements.
G. P. COOK

200. Universal high sensitivity photometer. For measuring light scattering, luminescence, transmittance and reflectance. G. Oster (*Anal. Chem.*, 1953, **25** [8], 1165–1169).—Details of a photometer of high sensitivity, including diagrams of the optical and electrical systems, are given. The optical components are readily interchangeable and coloured and neutral filters and polariser may be inserted in various combinations for both incident and observed

beams. The instrument is capable of measuring light intensities down to 20 microlamberts.
G. P. COOK

201. Differential refractometer. M. Sheffer, J. C. Hyde and H. J. Hadow, Assrs. to Min. Nat. Defence, Canada (U.S. Pat. 2,630,042, 3.3.53. Appl., 7.11.50).—A device for measuring the differences between the refractive indices of two liquids is illustrated and claimed.   O. POTTER

202. A modified Michelson refractometer. P. G. Guest and W. M. Simmons (*J. Sci. Instrum.*, 1953, **30** [10], 361–362).—An additional mirror is incorporated in a Michelson interferometer so that the interfering beams are parallel for the greater portion of their paths.   G. SKIRROW

Thermal

203. A method of measuring the temperature at the surface of a metal tube. A. G. Monroe and H. A. S. Bristow (*J. Sci. Instrum.*, 1953, **30** [10], 385).—A shallow helical groove in the surface of the tube contains insulated Pt resistance wire embedded in "cold solder." This method is claimed to give better results than the embedded thermo-couple method.   G. SKIRROW

204. An electronic temperature controller. B. E. Noltingk and M. A. Snelling (*J. Sci. Instrum.*, 1953, **30** [10], 349–351).—A thermistor forms one arm of an a.c. (50 c.p.s.) Wheatstone bridge. The off-balance signal is fed, after amplification, to a phase-sensitive detector, which operates the heating circuit relay. Control of an eight gallon water-bath to ± 0·02° C over a period of weeks is claimed.
G. SKIRROW

205. Two new forms of melting point calorimeters for determining the purity of liquids of condensed gases. John T. Clarke, H. L. Johnston and W. De Sorbo (*Anal. Chem.*, 1953, **25** [8], 1156–1159).—A description of two calorimeters is given, which includes details of construction and operation. One calorimeter requires the use of a platinum resistance thermometer embedded in the sample and gives reliable results when the heat of fusion is known or when the cryoscopic constant is determined by addition of an impurity to the sample. The other calorimeter is more accurate and requires the use of a Cu block whose temperature is carefully controlled; with this calorimeter heats of fusion sufficiently accurate to determine cryoscopic constants were determined.   G. P. COOK

206. Method and apparatus for determining the freezing point of liquids. J. W. Dickey, Assr. to Bendix Aviation Corp. (U.S. Pat. 2,633,736, 7.4.53—Appl., 28.3.49).—A freezing chamber surrounding a thermometer, but sealed therefrom, has an opening for receiving a sample of the liquid to be tested by means of a receiver. A flexible diaphragm closing the receiver can be dilated and held dilated manually. Elastic means apply continuous pressure to the diaphragm when the dilating means is released. Manual control is used for circulating a freezing medium around the chamber. O. POTTER

207. Flash point apparatus. R. Matteson, Assr. to California Research Corp. (U.S. Pat. 2,627,745, 10.2.53—Appl., 16.6.50).—A test cup for the determination of the flash point of a volatile liquid by periodic exposure of a heated film of the liquid to an ignition source is claimed.   O. POTTER

208. A rapid method for the determination of the thermal conductivity of organic liquids for use in heat-transfer calculations. S. Baxter, H. A. Vodden and S. Davies (*J. Appl. Chem.*, 1953, **3** [10], 477–480).—The apparatus developed consists of two concentric brass cylinders around which the liquid flows. Heat is supplied to the liquid by a constantan coil inside the centre tube. The temp. drop across the annular space between the tubes is measured by a thermocouple with the ends embedded longitudinally in the tube walls. Rapid measurements can be made over a wide range of temp. The results for a number of pure liquids agree with recorded values. Data for tetra-octyl silicate are presented up to 220° C.   A. P. J. CHATER

209. Thermal analysis with recording of the volume of gases liberated. B. Ya. Teytelbaum and L. G. Berg (*J. Anal. Chem., U.S.S.R.*, 1953, **8**, 152–157).—Improvements in the apparatus of Berg (*Zavod. Lab.*, 1948, **14**, 1171) are described.   G. S. SMITH

210. Differential thermobalance: a new research tool. W. L. de Keyser (*Nature*, 1953, **172**, 364–365).—Two crucibles containing initially equal weights of material are suspended from a balance inside vertical tubular furnaces, which are gradually heated so that the temp. of one is constantly 4° above that of the other. By means of an optical system the difference in weight is photographically recorded; the record is then equivalent to a plot of the rate of loss of weight against temp. This is more convenient than a plot of the integral weight loss, and enables small changes to be observed and measured more accurately. An example is given of the dehydration of a mixture of kaolin with a small quantity of diaspore.   H. P. PAGET

211. Improvements relating to temperature measuring apparatus. Sangamo Weston, Ltd. (Brit. Pat. 696,155, 30.3.51 U.S., 22.6.50).—The measurement of the average temperature of a liquid regardless of the actual depth of the liquid is carried out by a temperature measuring element supported by a movable tubular member or similar holder and immersed and extended throughout the depth of the liquid for all levels. A temperature indicator is provided, responsive to the measuring element which may comprise a plurality of coils secured on a pantograph mechanism; but various known means of indicating the temperature may be used or for giving a signal upon departure of the average temperature from a predetermined figure. The temperature sensing device may be pivotally mounted adjacent the bottom of the liquid and have a float secured to the upper end. The mechanism is described in detail, and various arrangements cited, in conjunction with a series of diagrams.
J. N. T. LINTOTT

Electrical

212. A stable source of high voltage. L. U. Hibbard and D. E. Caro (*J. Sci. Instrum.*, 1953, **30** [10], 378–380).—The unit described provides a voltage reference in the 1000 V region. Stability of 0·01 per cent. over a day is claimed. Negligible effect is produced by stray magnetic fields.
G. SKIRROW

213. Potentiometric polarography. Controlled current scanning. Ralph N. Adams, C. N. Reilley and N. H. Furman (*Anal. Chem.*, 1953, **25** [8], 1160–1164).—A method for obtaining polarograms at solid electrodes is described. The conventional technique is reversed, manually controlled current scanning and observation of the resulting voltage with a continuous indicating pH meter being applied. *iR* drop corrections are not necessary in this method and linear concentration - diffusion current conditions exist. The method, when applied to the determination of Fe^{III} in synthetic mixtures containing Fe$_2$O$_3$, gives results within 1 per cent. of the true value.   G. P. COOK

214. Method and apparatus for the determination of electrical conductivity. O. H. Drager (Brit. Pat. 695,568, 13.9.50; Ger., 21.9.49).—The method, which is claimed to be specially applicable to measuring the conductivity of electrolytes, materials of low conductivity, and substances sealed in ampoules, consists in placing the material opposite a constant dielectric and at least one condenser plate so connected to a measuring instrument that a displacement current flows through the dielectric and a conductivity current flows through the material. The complex alternating current resistance is measured by the instrument. The general method may be subjected to minor stated variations.
J. N. T. LINTOTT

215. An improved Rankine magnetic susceptibility balance for use in free radical determinations. J. O'M. Bockris and D. F. Parsons (*J. Sci. Instrum.*, 1953, **30** [10], 362–363).—An instrument is described for the determination of free radicles of lifetime longer than one min. and in concn. greater than 0·0004 *M*.   G. SKIRROW

216. A recording fluxmeter. R. S. Tebble (*J. Sci. Instrum.*, 1953, **30** [10], 369–371).—The secondary coil of a mutual inductance is attached to the suspended coil of a Grassot fluxmeter and a deflection of the fluxmeter needle is recorded as a change in a voltage induced in the mutual inductance system. Part of the voltage is fed back to eliminate the torsion effect of the fluxmeter suspension. The output voltage can be used to operate a recorder or oscillograph.   G. SKIRROW

217. 60-Cycle source for operation of laboratory timers. W. J. Biermann and N. Weber (*Anal. Chem.*, 1953, **25** [8], 1284–1285).—By using the circuit shown for driving a 5-watt synchronous motor, time intervals can be measured to within 0·001 per cent. of the intervals between time signals broadcast by the National Bureau of Standards. O. M. JONES

218. An instrument for measuring speed fluctuations, and a method of calibrating such instruments. A. Swindells (*J. Sci. Instrum.*, 1953, **30** [10], 364–365).—An a.c. tachogenerator and amplifier and filter equipment are used to measure fluctuations in low rotational and linear speeds.   G. SKIRROW

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.		micro-litre	μl
ampere	amp.		micron	μ
Ångstrom unit	A		milliampere	mA
anhydrous	anhyd.		milligram	mg
approximate, -ly	approx.		millilitre	ml
aqueous	aq.		millimetre	mm
atmospher-e, -ic	atm.		millivolt	mV
atomic	at.		minimum	min.
boiling-point	b.p.		minute (time)	min.
British thermal unit	B.Th.U.		molar (concentration)	M
calculated	(calc.)		molecul-e, -ar	mol.
calorie (large)	kg-cal.		normal (concentration)	N
calorie (small)	g-cal.		number	no.
centimetre	cm		observed	(obs.)
coefficient	coeff.		organic	org.
concentrated	conc.		ounce	oz.
concentration	concn.		part	pt.
constant	const.		patent	pat.
corrected	(corr.)		parts per million	p.p.m.
critical	crit.		per cent. wt. in wt.	per cent. w/w
crystalline	} cryst.		per cent. wt. in vol.	per cent. w/v
crystallised			per cent. vol. in vol.	per cent. v/v
cubic	cu.		potential difference	p.d.
current density	c.d.		pound	lb
cycles per second	c.p.s.		precipitate	ppt.
decompos -ing, -ition	(decomp.)		precipitated	pptd.
density	ρ		precipitating	pptg.
density, relative	d or wt. per ml		precipitation	pptn.
derivative	deriv.		preparation	prep.
dilute	dil.		qualitative, -ly	qual.
direct current	d.c.		quantitative, -ly	quant.
distilled	dist.		recrystallised	recryst.
electromotive force	e.m.f.		refractive index	$n\lambda^t$
electron-volt	eV		relative humidity	R.H.
equivalent	equiv.		revolutions per minute	r.p.m.
experiment, -al	expt.		saponification value	sap. val.
gram	g		saturated calomel electrode	S.C.E.
gram-molecule	mole		second (time)	sec.
half-wave potential	$E_{\frac{1}{2}}$		soluble	sol.
horse-power	h.p.		solution	soln.
hour	hr.		specific gravity	sp. gr.
hydrogen ion concentration	[H$\cdot$]		specific rotation	$[\alpha]\lambda^t$
hydrogen ion exponent	pH		square centimetre	sq. cm
inch	in.		standard temperature and	
indefinite	indef.		pressure	s.t.p.
infra-red	i.r.		temperature	temp.
insoluble	insol.		ultra-violet	u.v.
kilogram	kg		vapour density	v.d.
kilovolt	kV		vapour pressure	v.p.
kilowatt	kW		volt	V
liquid	liq.		volume	vol.
maxim -um, -a	max.		watt	W
melting-point	m.p.		wavelength	λ
microgram	μg		weight	wt.

In addition the following symbols are used—

greater than	>	less than	<
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\approx$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, e.g., Cu$\cdot\cdot$, Al$\cdot\cdot\cdot$, Cl$'$, SO$_4''$. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, e.g., ferric iron becomes Fe^{III} and cuprous copper Cu^I.

ANALYTICAL ABSTRACTS

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